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**Mesoarchaean (2820 Ma) high-pressure mafic granulite at Uauá, São  
Francisco Craton, Brazil, and its potential significance for the  
assembly of Archaean supercratons**

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## 26    **Abstract**

27    High pressure (HP) granulites of regional scale form as a result of tectonic events that  
28    lead to crustal thickening or subduction of the crust into the mantle. Most HP granulites  
29    are Phanerozoic, a few are Proterozoic, and Archaean HP granulites are even scarcer.  
30    Here we present field relationships, mineral chemistry and zircon U-Pb ages, Hf isotope  
31    data and trace elements data for the mafic granulite and associated rocks of the Uauá  
32    terrane, São Francisco Craton, Brazil, as evidence for the likely existence of a thick  
33    continental crust in the Mesoarchaeoan/Neoarchaeoan transition. The HP mafic granulite  
34    occurs as lensoid bodies within shallow dipping diorite to leucodiorite gneisses. Small  
35    scale layering between mafic granulite and diorite gneiss indicate these rocks are  
36    cogenetic. Garnet-clinopyroxene pairs with quartz, zircon, ilmenite, plagioclase, and  
37    clinopyroxene inclusions in garnet characterize the HP assemblage. Garnet  
38    porphyroblasts also show opx-cpx-plag symplectite coronas, which coupled with  
39    hornblende and plagioclase define PT conditions to lower grade granulite and  
40    amphibolite facies. Microprobe data combined with phase equilibria modelling  
41    (pseudosections) indicate 16-18 kbar and 930-960 °C for the peak HP assemblage, and  
42    6.2-7.0 kbar and 660-760°C for lower pressure granulite to amphibolite facies  
43    symplectite coronas. Metamorphic zircon rims in equilibrium with garnet have  
44    SHRIMP ages of  $2819 \pm 14$ , and igneous zircon cores of  $3127 \pm 14$  Ma. The cores of  
45    zircon in associated gneiss samples have U-Pb ages between  $3090 \pm 13$  Ma (LA-ICP-  
46    MS) and  $3125 \pm 15$  Ma (SHRIMP) with age cluster at  $3120 \pm 6$  Ma.  $\epsilon_{\text{Hf}(t)}$  values on  
47    igneous zircon of the HP mafic granulite are slightly positive whereas metamorphic  
48    zircon rims are negative. The associated diorite gneiss invariably yielded negative  
49    zircon  $\epsilon_{\text{Hf}(t)}$  values. Trondhjemite sheets intrusive in the mafic granulite are ca. 20 m.y.  
50    younger ( $2794 \pm 13$  Ma) than the host granulite. We interpret the igneous protoliths of  
51    the mafic granulite and leucodiorite gneiss as a single igneous complex emplaced in the

continental crust, later deformed and metamorphosed by contraction and crustal thickening during collision of blocks/cratons to form Earth's first supercratons by the end of the Mesoarchaeon.

**Keywords:** Archaean High-Pressure Mafic Granulite, Thermobarometry, Pseudosections, U-Pb Zircon Geochronology, Tectonics, São Francisco Craton

## **1. Introduction**

Most exposed Archaean crust is composed of low-grade granite-greenstone terranes and high-grade granulite-gneiss terranes (e.g. Windley 1995; Condie 2011). Whereas the low-grade terranes have been well studied (e.g. Polat al., 2009; Dilek and Furnes 2014), the high-grade metamorphic terranes are less well known, because of their more intense deformation and metamorphism, and because of the scarcity of modern analogues/equivalents (Windley and Garde, 2009). Today, most high-grade terranes have an amphibolite or granulite facies metamorphic grade, many amphibolite facies terranes retain evidence of retrogression from the granulite facies, and many have undergone major imbricate thrusting and tectonic intercalation, followed by several phases of isoclinal to tight folding and granulite-amphibolite facies metamorphism (e.g. Myers, 1976; Sajejev and Santosh, 2006). Thus, deep crustal sections of orogenic belts are still poorly understood compared with upper crustal sections (Polat et al., 2009; Dumond et al., 2010; Dilek and Furnes, 2014).

High-pressure (HP) granulites occur as layers and lenses in high-grade granulite-gneiss terranes; they form as a result of crustal thickening by collision (Brown, 2009), subduction of the crust into the mantle (O'Brien and Rötzler, 2003), or cooling from magmatic conditions to HP metamorphic conditions at the base of island arc complexes (De Paoli et al., 2009; Brown, 2009). The minimum PT conditions for HP granulite

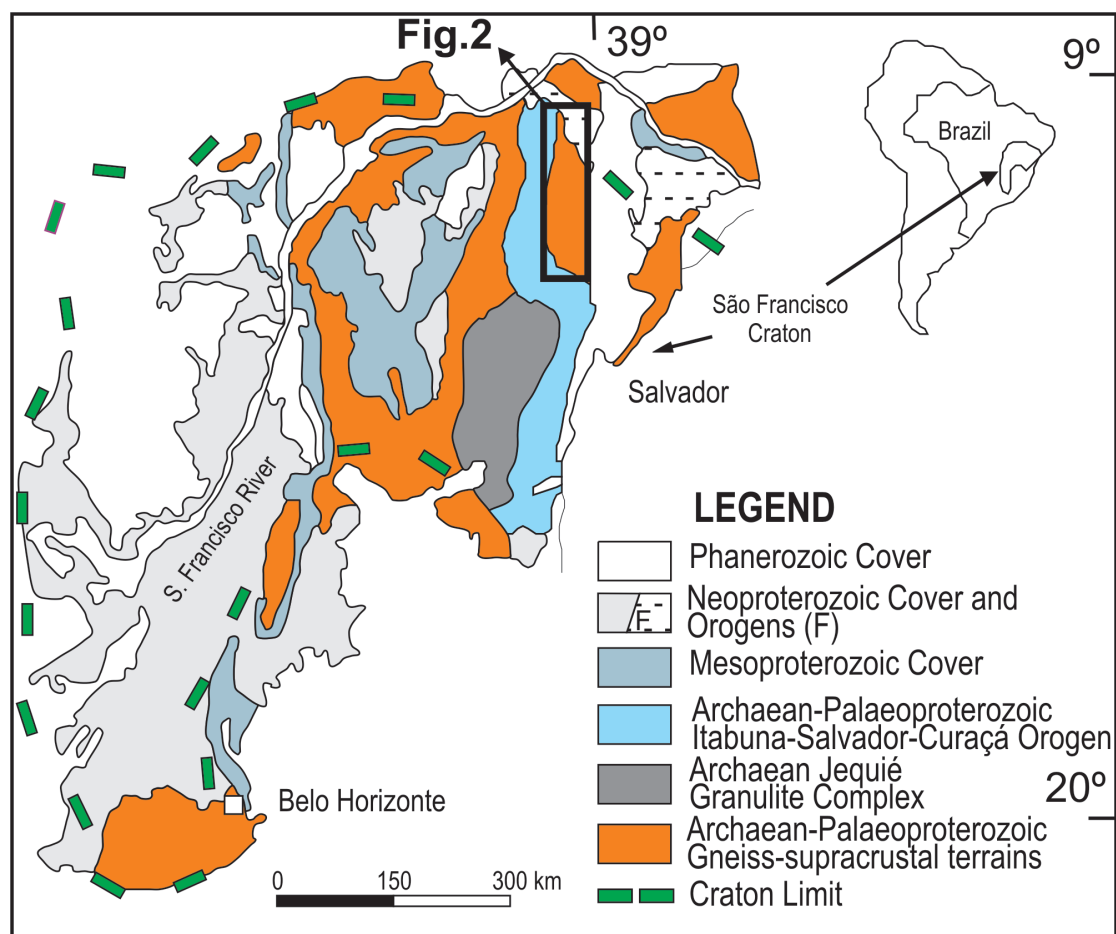


formation range from 11.5 kbar and 680°C (Oh and Liou, 1988) to 9 kbar and 620°C (Sajeev and Santosh, 2006). Most HP granulites are Phanerozoic, there are fewer well-documented occurrences of early to late Proterozoic age (Baldwin et al., 2006), and Archaean HP granulites are even scarcer. The aim of this paper is to report a new discovery of ca. 2.8 Ga HP mafic granulites in the Uauá terrane, São Francisco Craton, Brazil, in order to provide much needed information on the make-up and evolution of the lower crust of orogenic belts.

## **2. Geological Setting**

The São Francisco Craton (SFC) in eastern Brazil (Fig. 1) is surrounded by Neoproterozoic orogens (Araçuaí, Brasília, Riacho do Pontal, Rio Preto, and Sergipano).

The basement rocks of the SFC are dominated by Archaean to Palaeoproterozoic migmatites, amphibolite to granulite-grade gneisses, and granite-greenstones (Teixeira et al., 2017). Meso-Neoproterozoic sedimentary rocks of the Espinhaço and São Francisco Supergroups cover those rocks. The SFC, which aggregated during the Archaean and Proterozoic mainly in the interval 2.1-1.8 Ga, is made up of several greenstone belts (e.g. the Rio Capim, Rio Itapicuru, Mundo Novo, Umburanas, and Rio das Velhas) and microcontinental blocks (e.g. the Gavião, Jequié, Itabuna-Salvador-Curaçá orogen and Serrinha). Relevant here is the Serrinha Block, which contains the Uauá high-pressure mafic granulites.



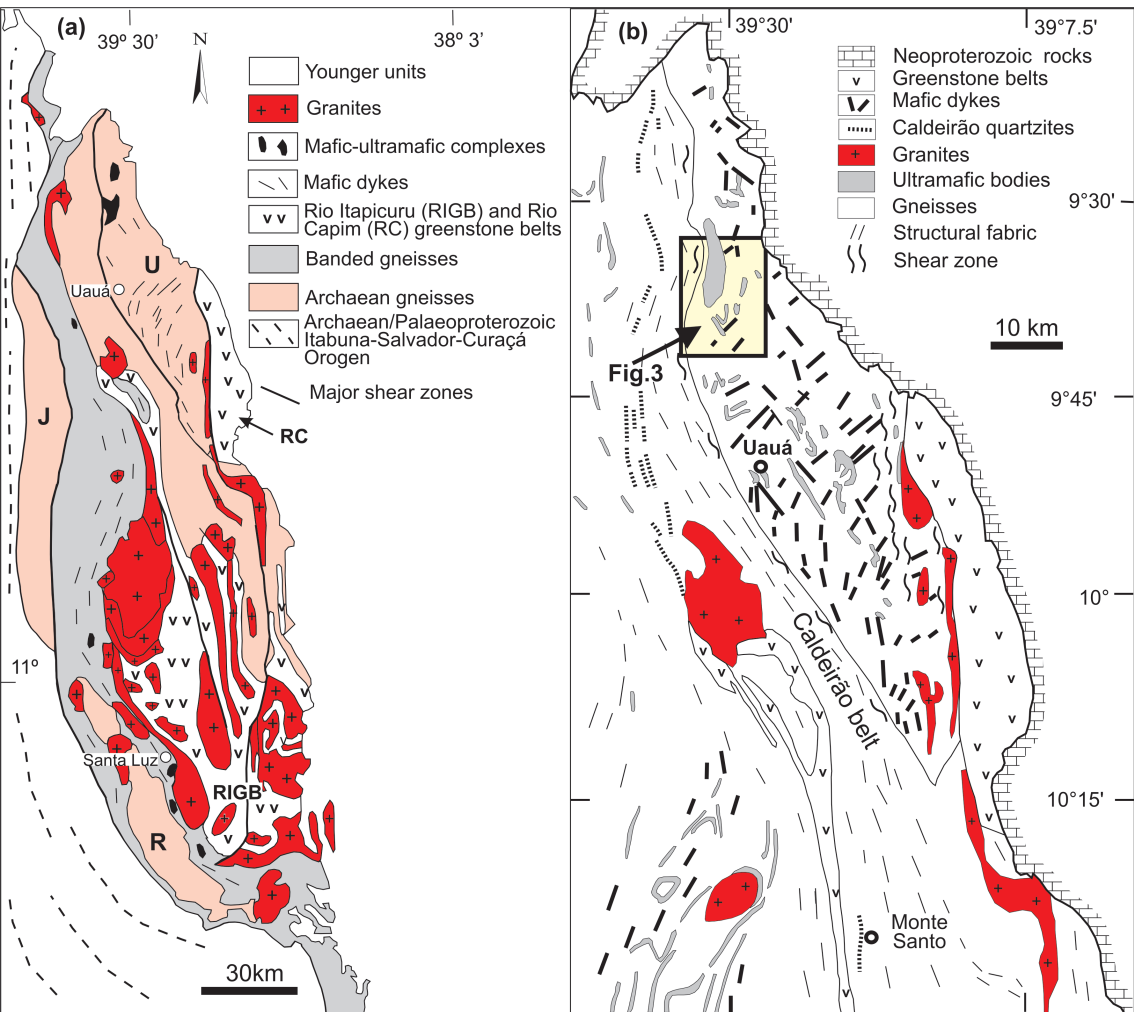
105

106 **Fig. 1.** The main geologic units of the São Francisco Craton (modified from Oliveira et  
 107 al., 2004). The box indicates the Serrinha Block that contains the Uauá high-pressure  
 108 mafic granulite.

109

110 The Serrinha Block, or microcontinent (Fig. 2a) is composed of amphibolite to  
 111 granulite facies grey gneisses of the Tonalite-Trondhjemite-Granodiorite (TTG) suites,  
 112 migmatitic grey gneisses, banded gneisses, marbles, BIFs, mafic dykes and mafic-  
 113 ultramafic complexes; the banded gneisses are the end-products of deformation of the  
 114 migmatitic TTG gneisses and mafic dykes (Oliveira et al., 2010). These rocks are in  
 115 tectonic contact with the supracrustal Palaeoproterozoic Rio Itapicuru and Rio Capim  
 116 greenstone-granite belts. The grey gneisses have ages from 3151 Ma to 2933 Ma and  
 117 crop out continuously close to the towns of Retirolândia and Uauá, and along the

Jacurici river (R, U and J, respectively in Fig. 2a); they may be terranes of the Serrinha Block (Oliveira et al., 2010). The Rio Itapicuru low-grade supracrustal greenstone belt (RIGB in Fig. 2a) is about 180 km long, 30 km wide and has three lithostratigraphic subdivisions: (i) a basal mafic volcanic unit composed of massive and pillowed basaltic flows intercalated with chert, banded iron-formation, and carbonaceous shale; (ii) an intermediate to felsic volcanic unit with meta-dacites, meta-andesites and meta-pyroclastic rocks, and (iii) a meta-sedimentary pelitic-psammitic unit mainly composed of meta-pelites and minor chemical sedimentary rocks. Zircon and xenotime U-Pb geochronological data indicate this belt formed between 2170 Ma and 2080 Ma (Mello et al., 2006, Oliveira et al., 2010). The high-pressure mafic granulite occur in the Uauá terrane, which is bounded by the Rio Capim greenstone belt and the Caldeirão shear belt, the geological details of which are given below.



**Fig. 2.** The Serrinha Block and its main geological units. a) Letters show the Mesoarchaeon grey gneisses, or terranes of Retirolândia (R), Jacurici (J), and Uauá (U). The banded gneisses (in gray) are the metamorphic product of collision between the Mesoarchaeon terranes and the Palaeoproterozoic greenstone belts (RIGB and RC); after Oliveira et al. (2010). b) Detail of the Uauá terrane bounded by shear zones with the Caldeirão belt (west) and the Rio Capim greenstone belt (east). Fig. 3 shows location of the high-pressure mafic granulite.

### 2.1. The Uauá Terrane

The Uauá terrane (Fig. 2b; Uauá pronounced like the Canadian town “Wawa”) was displaced from south to north (present-day coordinates) during Palaeoproterozoic oblique collision (Oliveira et al., 2004; Oliveira et al., 2010, Oliveira, 2011). It is bordered to the west by the Archaean-Palaeoproterozoic Caldeirão shear belt and to the east by the Palaeoproterozoic Rio Capim greenstone belt. Neoproterozoic continental shelf metasedimentary rocks of the Sergipano orogen cover all these units.

The Uauá terrane consists mostly of NW-trending banded gneisses of unknown age, layered anorthosite-peridotite-diorite complexes, and tonalite-granodiorite bodies (Oliveira, 2011), most of which were metamorphosed under granulite facies conditions and later retrogressed to amphibolite grade. Mesoarchaeon ages are widespread in the Uauá terrane. Anorthosites of the Lagoa da Vaca layered anorthosite complex have a  $3161 \pm 65$  Ma whole-rock Pb-Pb isochron, and orthogranulites have a zircon Pb-evaporation age of  $3072 \pm 20$  Ma (Paixão and Oliveira, 1998). Granulite and a gneissic granodiorite near Uauá town contain  $2933 \pm 3$  Ma and  $2991 \pm 22$  Ma zircons, respectively (Oliveira et al., 2002). The following rocks have zircon LA-ICP-MS ages: near Uauá there are ca. 2960 Ma folded sanukitoid sheets, 2955 Ma granodiorite

gneisses, and 3075 Ma gabbro lenses, stacked in N-verging nappes, whereas east of Uauá the rocks are less deformed and mainly represented by the ca. 3098-3079 Ma calc-alkaline Capim Tonalite, the 3085 Ma Caratacá porphyritic tonalite-granodiorite, and grey gneisses (Oliveira et al., 2016).

Two mafic dyke swarms intrude the Archaean gneisses of the Uauá terrane (Fig. 2b); a NW-trending swarm of metamorphosed tholeiite dykes and unmetamorphosed or weakly metamorphosed 2726 Ma norite-pyroxenite dykes, and a NE-trending swarm of unmetamorphosed 2624 Ma tholeiite dykes (Oliveira et al., 2013). Crosscutting relations and drag folds indicate the NE-trending dykes are the younger (Oliveira, 2011; Oliveira et al., 2013).

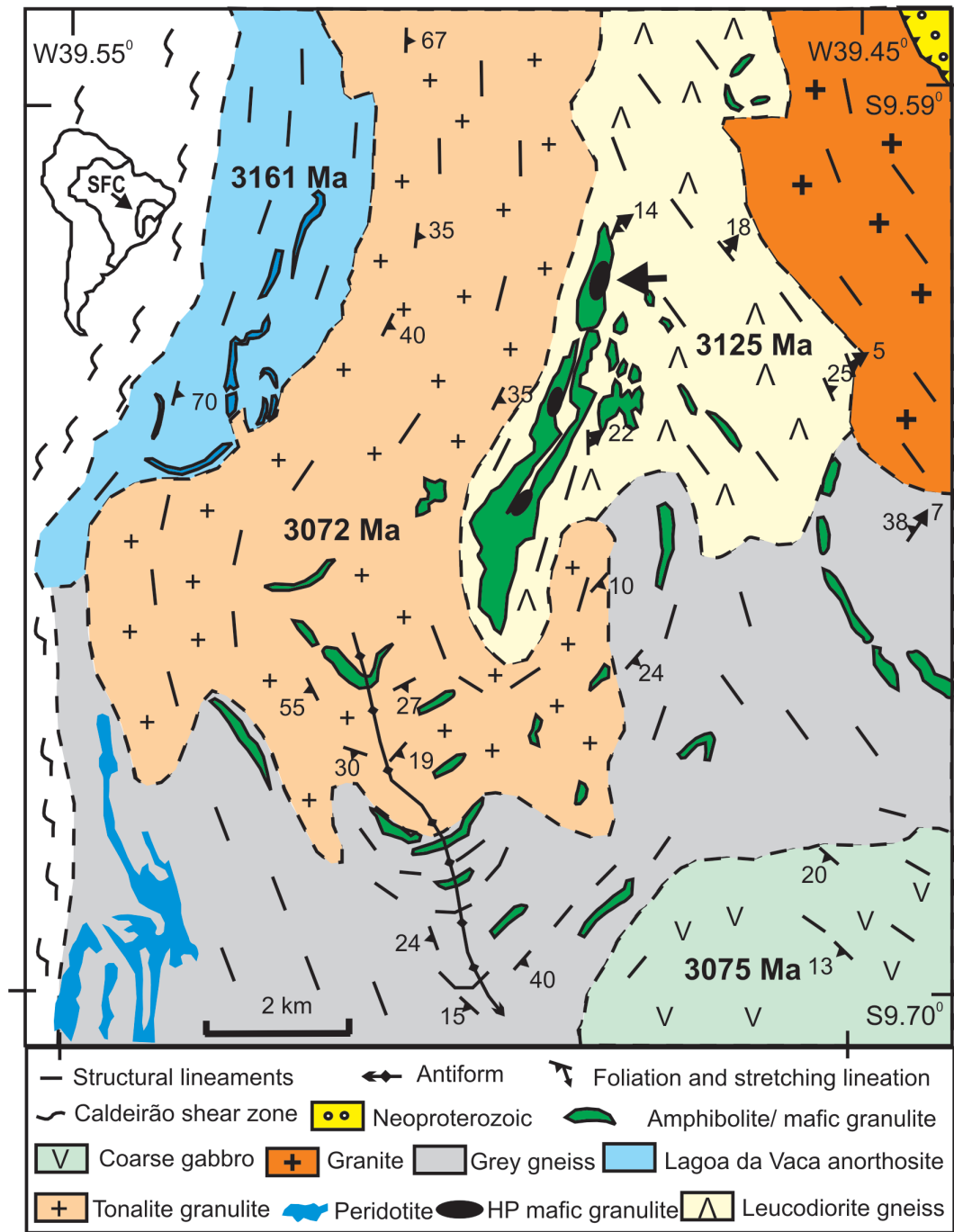
The 10 km-wide Caldeirão shear belt (Fig. 2b) comprises steeply dipping quartzites, sillimanite-cordierite-garnet gneisses, granodiorite gneisses, mafic rocks and migmatites, all metamorphosed in the amphibolite facies; the granodiorite gneiss protoliths have a SHRIMP U-Pb age of 3150 Ma (Oliveira et al., 2002). To the south, the shear belt is dismembered into narrow sinistral strike-slip shear zones, one of which continues for over 150 km across the Rio Itapicuru greenstone belt. The boundary of this shear belt to the Uauá terrane is gradational and marked by refolding of older structures, by granite and pegmatite intrusions, and by shear zones. Detrital zircon grains in quartzites and syn-deformational titanite in mafic dykes constrain the quartzite protolith depositional age to a maximum of 2700 Ma and a regional metamorphic age to between 2039 and 2077 Ma (Oliveira et al., 2000, 2002).

The Rio Capim greenstone belt (Fig. 2) is a 10-km wide, 80-km long, N/NW-trending belt of deformed and metamorphosed mafic to felsic volcanic and associated pelitic rocks, intruded by plutons ranging in composition from gabbro/diorite to granite (Oliveira et al., 2011). In this belt the metamorphic grade increases from greenschist facies in the west to granulite in the east. Zircon U-Pb ages of the felsic volcanic, diorite

and granite fall in the range 2148-2128 Ma (Oliveira et al., 2011). The contact of the Rio Capim greenstone belt and the Uauá terrane is occupied by the 20-500 meter-wide, NS-trending, upright Galo do Ouro dextral shear zone (Oliveira, 2011).

### ***2.1.1. The mafic granulite containing HP relics***

The mafic granulite occurs as dozen to hundred m-wide and several hundred m- to km-long lensoid bodies within quartz diorite, leucodiorite to granodiorite gneisses (Fig. 3). The black arrow in Fig. 3 indicates the largest and most continuous outcrop of mafic granulite containing high-pressure relics, which forms a swarm of narrow massive to layered bands (Fig. 4a), massive lenses with disseminated garnet (Fig. 4b), garnet-rich pods (Fig. 4c), and garnet-rich boudins (Fig. 4d) within mafic granulite partially retrogressed to amphibolite facies. The contact between mafic granulite and leucodiorite gneiss is gradational (Fig. 5a) suggesting that their protoliths were parts of layered complexes. Narrow trondhjemite sheets intrude the mafic granulite as evidenced by their contact paralleling (Fig. 5b) or crosscutting (Fig 5c) the host mafic granulite foliation. The associated leucodiorite/diorite gneisses dip shallowly eastwards and have a mineral stretching lineation that plunges shallowly to E-NE (Fig. 3). Locally, bands of leucodiorite gneiss are displaced along flat-ramp thrust faults indicating late contraction towards the W-SW (Fig. 5d). To the west both mafic granulite and host leucodiorite gneiss are in contact with a large body of magnetite-rich, quartz-poor, tonalite granulite, which in turn is in contact to the west with the ca. 3.16 Ga Lagoa da Vaca layered anorthosite (Paixão and Oliveira, 1998). To the east, the leucodiorite-granodiorite gneiss is intruded by granites and to the south it is tectonically imbricated with grey gneisses (Fig. 3).



**Fig. 3.** Geological map with the main occurrences of mafic granulite. Ages of rock units are from Paixão and Oliveira (1998), Oliveira et al. (2016) and this work. Black arrow indicates the largest outcrop of mafic granulite where the samples for this study were collected (e.g. E0182).





213

214 **Fig. 4.** Field features of the mafic granulite. a) Massive to layered mafic granulite; b)

215 disseminated garnet in mafic granulite; c) garnet-rich mafic granulite within

216 retrogressed amphibole-bearing mafic granulite; d) layers and boudin of garnet-rich

217 mafic granulite within more felsic, foliated retrogressed amphibole-bearing mafic

218 granulite. 15 cm pen and 25 mm coin for scale.

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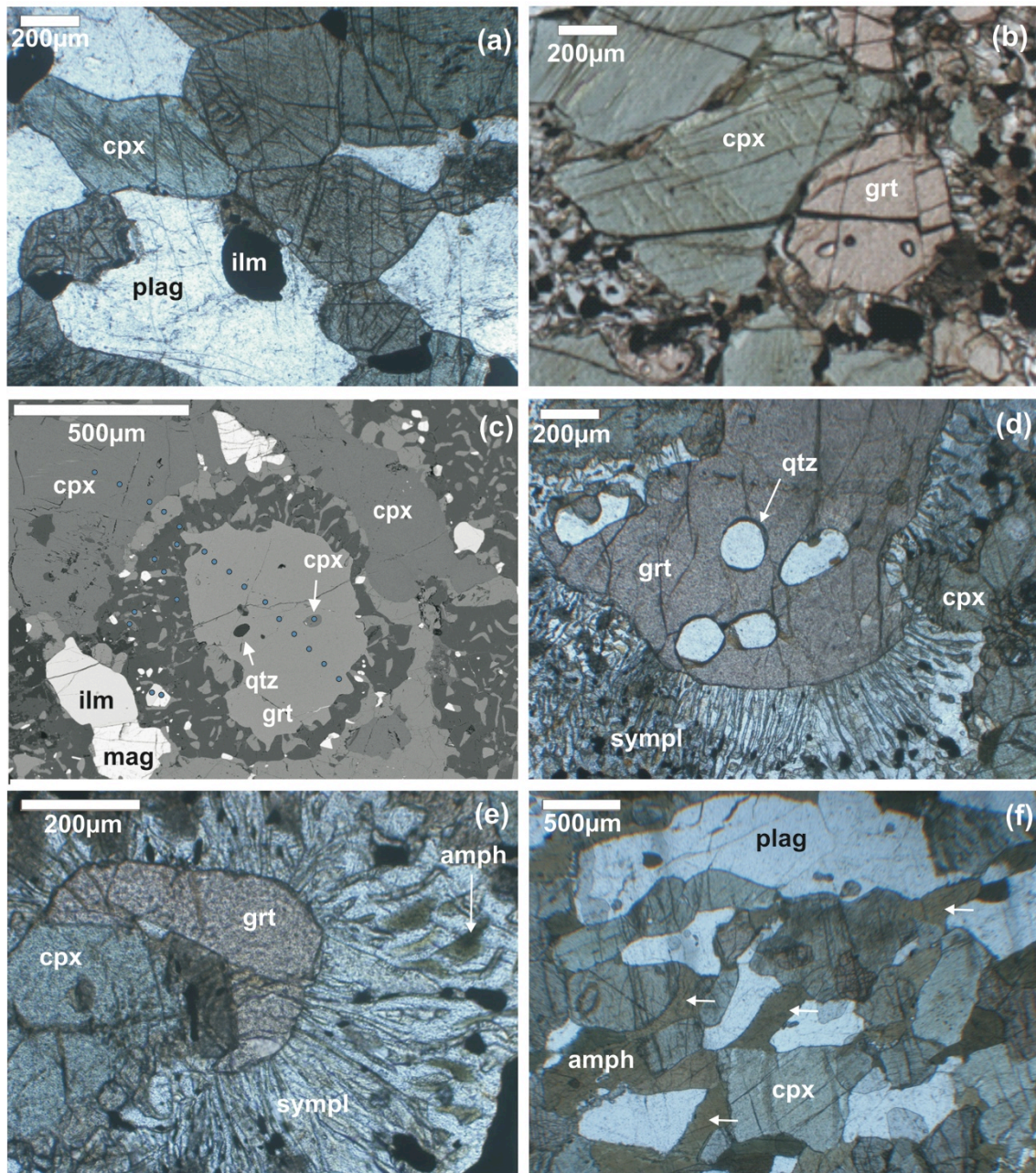
**Fig. 5.** Field relationships of the mafic granulite and associated leucodiorite-granodiorite gneiss. a) Gradational/layered contact between mafic granulite (towards figure top) and leucodiorite gneiss (towards figure bottom) suggesting that the protoliths of both mafic granulite and leucodiorite gneiss were cogenetic igneous rocks; b) younger trondhjemite sheets (white arrows) intrusive along the mafic granulite foliation; c) narrow trondhjemite offshoot (white arrow) crosscuts the mafic granulite foliation; d) shallow-dipping diorite/leucodiorite gneiss with thrust fault immediately above white arrow indicating top-to-SW thrusting and crustal thickening. Hammer, notebook or 15 cm pen for scale.

### 2.1.2. Petrography of the mafic granulite

The mafic granulite shows granoblastic textures and medium-to coarse grain size. It is composed of clinopyroxene, plagioclase and minor ilmenite and quartz (Fig. 6a). Clinopyroxene occurs as aggregates with grains up to 1 mm and is diopsitic/augitic with  $XMg \sim 0.6$  and low amounts of Na (0.02-0.03 a.p.f.u.) and Al (0.08-0.11 a.p.f.u.).

Associated plagioclase is nearly 1 mm long and andesine to labradorite in composition (An 40-60). The high-pressure granulite facies assemblage is characterized by garnet and clinopyroxene (Fig. 6b), less often plagioclase, ilmenite and quartz. Garnet porphyroblasts (up to cm size) are Fe-rich (Alm60) and contain inclusions of clinopyroxene, quartz and plagioclase (Fig. 6c,d). Garnet profiles show decreased pyrope but increased spessartine component from core to rim (see Fig. 7 in Section 4.1). Garnet grains are surrounded by symplectites of orthopyroxene + plagioclase (Fig. 6d,e), indicating retrogression to lower pressure granulite facies conditions. The retrogression continued down to amphibolite facies conditions as indicated by minor green amphibole as part of the symplectite intergrowth (Fig. 6e) and as hornblende blades aligned along the foliation plane (Fig. 6f). Symplectitic orthopyroxene occurs in small 20-25  $\mu\text{m}$  wide to lesser than 300  $\mu\text{m}$  long grains around garnet and has  $\text{XMg} \sim 0.4-0.3$ . Symplectitic plagioclase is more calcic (An 65-73) than the groundmass ones. Opaque minerals are predominantly ilmenite and, rarely, magnetite. However, magnetite is frequent in the symplectites. Neither orthopyroxene nor hornblende are present as inclusion in garnet or in equilibrium with it and are therefore not interpreted to be part of the high-pressure assemblage.





**Fig. 6.** Textures and mineralogy of the Uauá mafic granulite EO182. In all figures garnet typically has a corona of opx+plag. (a) mafic granulite with clinopyroxene, plagioclase and accessory ilmenite; (b) typical association of clinopyroxene and garnet as the high-pressure assemblage; (c) (SEM back scatter image - BSE) garnet contains cpx and quartz inclusions; (d) garnet with orthopyroxene+plagioclase symplectites and quartz inclusions; (e) Detail of garnet + clinopyroxene with opx+plag+magn+amphibole symplectite - note that amphibole forms more distal to garnet in the symplectite; (f) hornblende oriented along the granulite foliation. BSE bright minerals in (c) are

magnetite (white) and ilmenite (grey). In (b) garnet is in contact with cpx and in (d) garnet is separated from cpx by opx-plag symplectites. Small black circles in (c) represent electron microprobe analyses, discussed below.

### **3. Samples and Methods**

A list of all samples used for this study with geographic coordinates is given in supplementary file Table S1.

#### **3.1. Mineral Chemistry**

Mineral analyses of sample EO182 (polished thin sections EO182a and EO182b in Table S2) were acquired for pressure-temperature calculations with a Cameca SXFive microprobe at the Federal University of Rio Grande do Sul, Brazil. The instrument operated under the following routine conditions: a) for garnet: beam current of 25 nA, beam diameter of 1  $\mu\text{m}$ , and accelerating voltage of 15 kV; b) for plagioclase and pyroxenes: beam current of 15 nA, beam diameter of 1  $\mu\text{m}$ , and accelerating voltage of 15 kV. The standards used were those provided commercially by SPI Supplies for microanalysis (SPI 53 minerals > <http://www.2spi.com/item/02753-ab>). For this paper, the following mineral standards were used for instrument calibration: jadeite (Na), almandine (Si and Fe), pyrope (Mg and Al), orthoclase (K), diopside (Ca), chromite (Cr), rodonite (Mn), rutile (Ti). The crystals used were TAP (Na, Al, Si, Mg), PET (Ca, Cr, K, Ti) and LIF (Fe, Mn).

#### **3.2. Pressure-Temperature Quantitative Phase Diagrams (Pseudosection)**

Phase equilibria modelling on a given bulk-rock composition (pseudosections) can reveal stable mineral assemblages and their evolution in P-T space, and therefore yields results independent from geothermobarometry since a priori knowledge of

mineral compositions is not required. We used the Gibbs free energy minimization software Perple\_X 6.8.6 (Connolly, 2005) and an internally consistent thermodynamic data set (Holland and Powell, 2011) to calculate pseudosections in the P-T range of 5-20 kbar and 600-1100 °C. The whole-rock composition of sample EO182 was chosen as starting composition (caption for Fig. 10) since it preserved the peak granulite-facies assemblage with the lowest amount of amphibolite facies overprint. The oxides MnO and P<sub>2</sub>O<sub>5</sub> were not considered due to their relatively low abundances and/or an incomplete set of solid solution models defining the compositional space as: TiO<sub>2</sub>-K<sub>2</sub>O-Na<sub>2</sub>O-CaO-FeO-MgO-Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub>-O<sub>2</sub> (TiKNCFMASO). The ratio of ferrous to ferric iron has not been determined in the sample EO182, but based on the presence of ilmenite and rare magnetite, we assume a value of 0.2 wt% O<sub>2</sub> in the starting composition, which would be similar to values constrained by the quartz-fayalite-magnetite (QFM) buffer at granulite facies conditions. H<sub>2</sub>O, and consequently melting, is not considered in our calculations because we are interested in the high-grade granulite facies mineral assemblage, which is interpreted to be anhydrous based on petrographic constraints and the negative or very small LOI values for the mafic garnet granulite composition (see caption to Fig. 10). Furthermore, textural relationships in the field and thin sections indicate that the mafic rocks were overprinted to amphibolite facies conditions after peak granulite facies conditions. The following solid-solution models were used: olivine from Holland and Powell (1998), clinopyroxene (augite) from Green et al. (2016), orthopyroxene and garnet from White et al. (2014), plagioclase from Holland and Powell (2003), and ilmenite-hematite from White et al. (2000). Quartz, rutile and K-feldspar were assumed to be pure phases. Titanite was excluded, as it has not been detected in the samples. The sum of all considered oxides is normalized to 100% by Perple\_X at every pressure-temperature condition.

In addition to the simplifications of the compositional space, extra uncertainties derive from uncertainties in physical properties of end-member in the thermodynamic data set, uncertainties in the formulation of activity-composition relationships, analytical errors, errors in the estimation of the oxidation state, the assumption of equilibrium conditions, and natural petrographic variation in the samples (e.g. Palin et al., 2016). Especially the modelled stability of minor phases such as rutile and quartz are sensitive to uncertainties in bulk composition and solid solution models, which is why they were not considered for the determination of P-T conditions. Furthermore, the ilmenite-rutile transition is sensitive to the oxidation state of the bulk rock with the ilmenite stability increased towards higher pressures with increasing oxidation state. Also, selected solution models do not account for small amounts of titanium, that is likely present in both garnet and clinopyroxene resulting in a somewhat overestimated amount of titanium-bearing oxides such as ilmenite and rutile, which also increases the uncertainty.

### **3.3. Geochronology**

Samples EO182, 14EW-20, 15GE-61, and a polished slab of EO182 (EO182p), were selected for age dating on the SHRIMP (Table S3). Sample EO182 is a medium-grained, garnet-rich granulite that is representative high-pressure mafic granulite partially retrogressed to the amphibolite facies; the sample contains garnet porphyroblasts, clinopyroxene, and opx+plag symplectites around garnet (as shown in Fig. 6) within a groundmass of plagioclase and clinopyroxene with minor late hornblende. Samples 14EW-20 and 15GE-61 are leucodiorite gneisses associated with the HP mafic granulite; the two samples are mainly composed of hornblende, plagioclase with minor quartz and pyroxene, the latter is often partially replaced by hornblende, or hornblende + quartz.

Zircon U-Pb ages were obtained with a Sensitive High Resolution Ion Microprobe using the Perth Consortium SHRIMP II at Curtin University, Western Australia, based on the analytical procedures described by de Laeter and Kennedy (1998) and Kennedy and de Laeter (1994). Samples EO182, EO182p, 14EW-20 and 15GE-61 were analysed in three different sessions. A 25-30  $\mu$  m diameter spot was used with a mass-filtered  $O_2^-$ -primary beam of  $\sim 1.7$ -2.2 nA on zircons from samples EO182, 14EW-20 and 15GE-61, and a 10-15  $\mu$  m diameter spot was used with a mass-filtered  $O_2^-$ -primary beam of 1.2-1.5 nA on zircons from sample EO182p. Data for each spot was collected in sets of 6 scans on the zircons through the mass range of  $^{196}Zr_2O^+$ ,  $^{204}Pb^+$ , Background,  $^{206}Pb^+$ ,  $^{207}Pb^+$ ,  $^{208}Pb^+$ ,  $^{238}U^+$ ,  $^{248}ThO^+$  and  $^{254}UO^+$ . The  $^{206}Pb/^{238}U$  age standard and U-content standard used is BR266 (559 Ma; 903 ppm U; Stern 2001). The  $^{207}Pb/^{206}Pb$  standard used to monitor instrument-induced mass fractionation was OGC zircon (3467 $\pm$ 3 Ma; Stern et al. 2009). The  $^{207}Pb/^{206}Pb$  dates obtained on OGC zircons during the SHRIMP sessions matched the  $^{207}Pb/^{206}Pb$  standard age within uncertainty and no fractionated correction was warranted. The common Pb correction was based on the measured  $^{204}Pb$ -correction (Compston et al., 1984). The formula for Pb/U calibration is  $^{206}Pb^+/^{238}U^+ = a (^{254}UO^+/^{238}U^+)^b$  (Claoué-Long et al., 1995) using the parameter values of Black et al. (2003). The constant “a” is determined empirically from analyses of the standard during the analytical session. The programs SQUID II and Isoplot (Ludwig 2003, 2009) were used for data processing.

Leucodiorite gneiss EO178-D was analysed by LA-ICP-MS to identify concordant grains for further Hf isotope analysis. The results are shown in Table S3. The analytical work was performed at the Institute of Geosciences, University of Campinas-UNICAMP. Isotope data were acquired on an ICP-MS Element XR (Thermo Scientific), coupled with an Excite.193 (Photon Machines) laser ablation system, equipped with a two-volume HelEx ablation cell. The acquisition protocol followed

Navarro et al. (2015) with details in Verma et al. (2016); spot size is 25  $\mu\text{m}$ . Data were reduced off-line using the Iolite software (version 2.5) following the method of Paton et al. (2010), which involves subtraction of gas blank followed by downhole fractionation correction comparing with the behaviour of the 91500 reference zircon (Wiedenbeck et al., 1995). Peixe zircon standard (ID-TIMS age of  $564 \pm 4$  Ma; cf. Dickinson and Gehrels, 2003) was used to monitor the quality of the reduction procedures; during the analytical session 14 Peixe zircon analyses yielded the average age  $575 \pm 10$  Ma.

### 3.4. Trace element analyses of zircons

Rare earth element (REE) and other trace element data were obtained on dated zircons from sample EO182p (Table S4) by LA-ICP-MS at University of Campinas, in order to test for any major chemical changes in zircon during high-grade metamorphism. The REE data were acquired on an ICP-MS Element XR (Thermo Scientific), coupled with an Excite.193 (Photon Machines) laser ablation system, equipped with a two-volume HelEx ablation cell. The standard Nist612 was used for trace element calibration, and zircon standard 91500 (Wiedenbeck et al., 1995) was analysed (10 spots) as an unknown for analysis validation.

### 3.5. Hafnium isotope analyses of zircons

Concordant zircons from samples EO182 and EO178-D were analysed for Hafnium isotope data (Table S5) at Federal University of Ouro Preto, Brazil, using a multi-collector (MC)-ICP-MS Thermo-Scientific Neptune Plus system coupled to a Photon Machines 193 ( $\lambda = 193$  nm) ArF Excimer laser ablation system, and following the analytical procedures of Albert et al. (2016). During the two analytical sessions 7 and 11 spot analyses of GJ-1 zircon ( $^{176}\text{Hf}/^{177}\text{Hf} = 0.282000 \pm 0.00005$ ; Morel et al., 2008) yielded average  $^{176}\text{Hf}/^{177}\text{Hf}$  ratios of  $0.282005 \pm 0.00020$  and  $0.282004 \pm 0.00019$ , respectively.

### 3.6. Whole-rock geochemistry



Whole-rock geochemistry of mafic granulite EO182 (caption for Fig. 10) for phase equilibria modelling was undertaken at the University of Campinas with a Philips PW2100 X-ray fluorescence spectrometer using fusion beads for major and minor elements following the analytical procedures of Vendemiato and Enzweiler (2001).

## **4. Results**

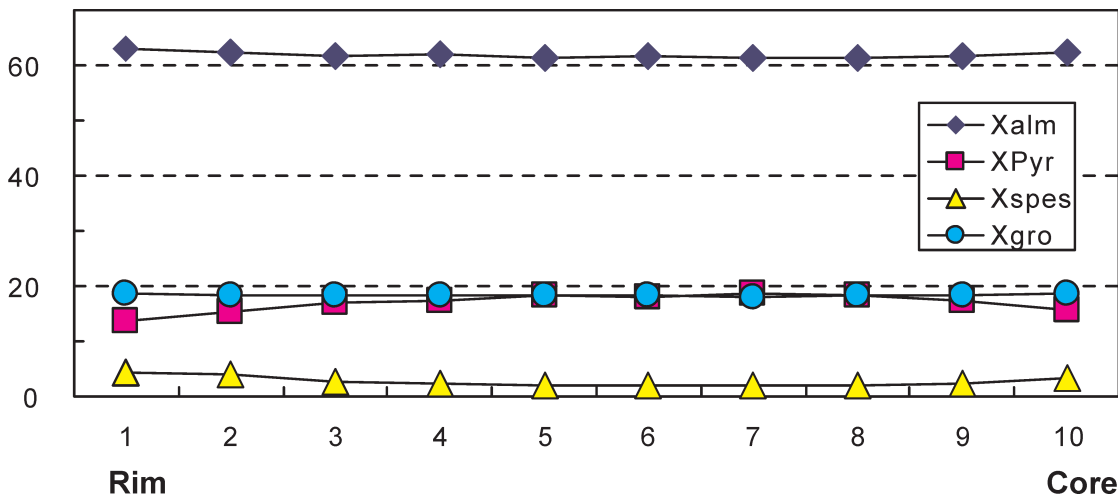
### **4.1. Metamorphic conditions: Thermobarometry**

Mineral compositions of two polished thin sections of sample EO182 (EO182a and EO182b) were analysed for thermobarometry calculations. Representative mineral compositions are presented in Table S2 (supplemental files).

Garnet-clinopyroxene pairs with quartz, zircon, ilmenite, plagioclase, and clinopyroxene inclusions in garnet define the high-pressure assemblage. Garnet porphyroblasts also show opx-cpx-plag symplectite coronas, which coupled with hornblende and plagioclase, define the retrogressive conditions to lower grade granulite and amphibolite facies.

Reaction textures preserved in the mafic granulite indicate that it experienced severe retrogression after peak metamorphic stage. Chemical zoning patterns of the garnet show clear decrease of  $X_{\text{Pyr}}$  and increase of  $X_{\text{Spes}}$  from core to rim. The variations of  $X_{\text{Alm}}$  and  $X_{\text{Gro}}$  are not so obvious and they seem to remain the same from core to rim (Fig. 7; transect shown in Fig. 6c). Such chemical zoning patterns of garnet belong to typical diffusion zoning patterns usually developed during retrograde metamorphism (Spear and Selverstone, 1983; Spear et al., 1990). In such kind of rocks, mineral compositions acquired during peak metamorphic stage (M2) are most likely found in the cores of garnet, cores of clinopyroxene, cores of plagioclase and cores of orthopyroxene. Compositions of rim parts of garnet grains and adjacent minerals, like

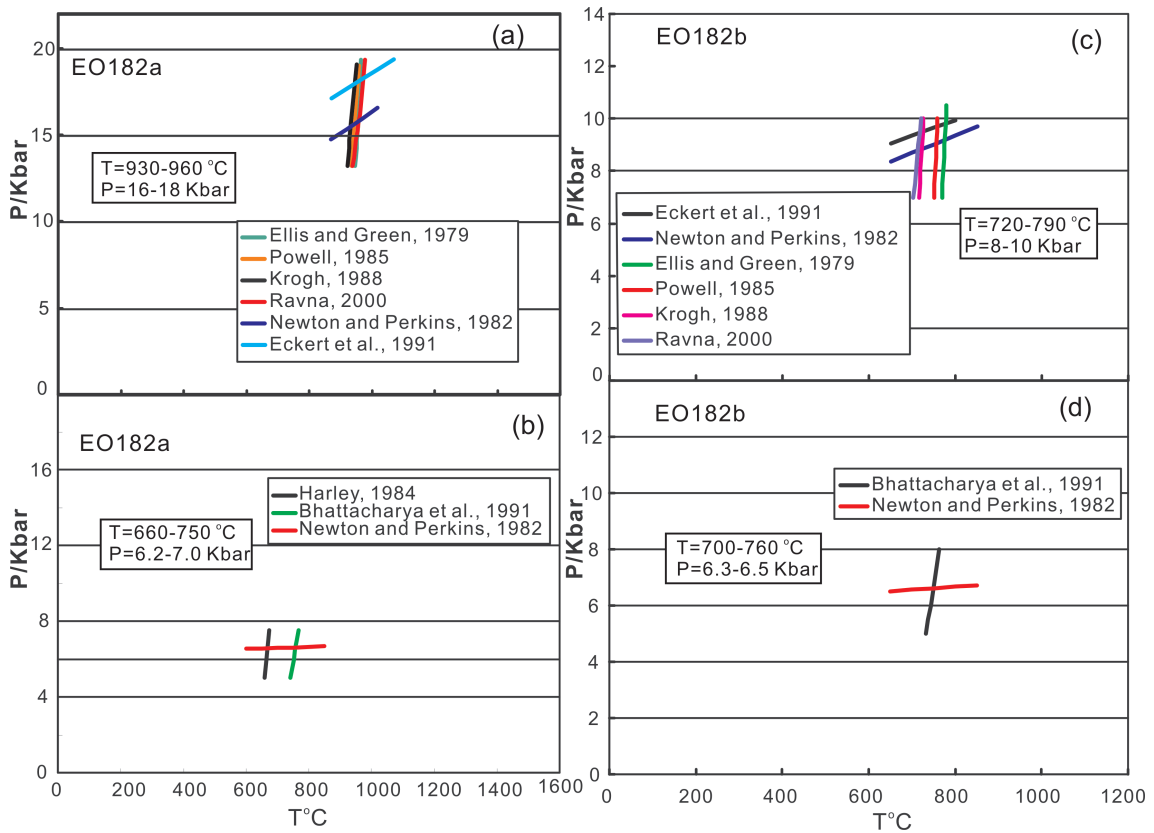
plagioclase, clinopyroxene, orthopyroxene and amphibole, are most likely related with retrograde stage (M3) metamorphism. As to the prograde metamorphic stage (M1), representative mineral compositions might not be able to survive the peak and retrograde metamorphic alteration. Therefore, in the following discussion, inferred mineral assemblages of M2 are garnet (core) + clinopyroxene (core) + plagioclase (core) + quartz while those of M3 are garnet (rim) + adjacent orthopyroxene + adjacent plagioclase.



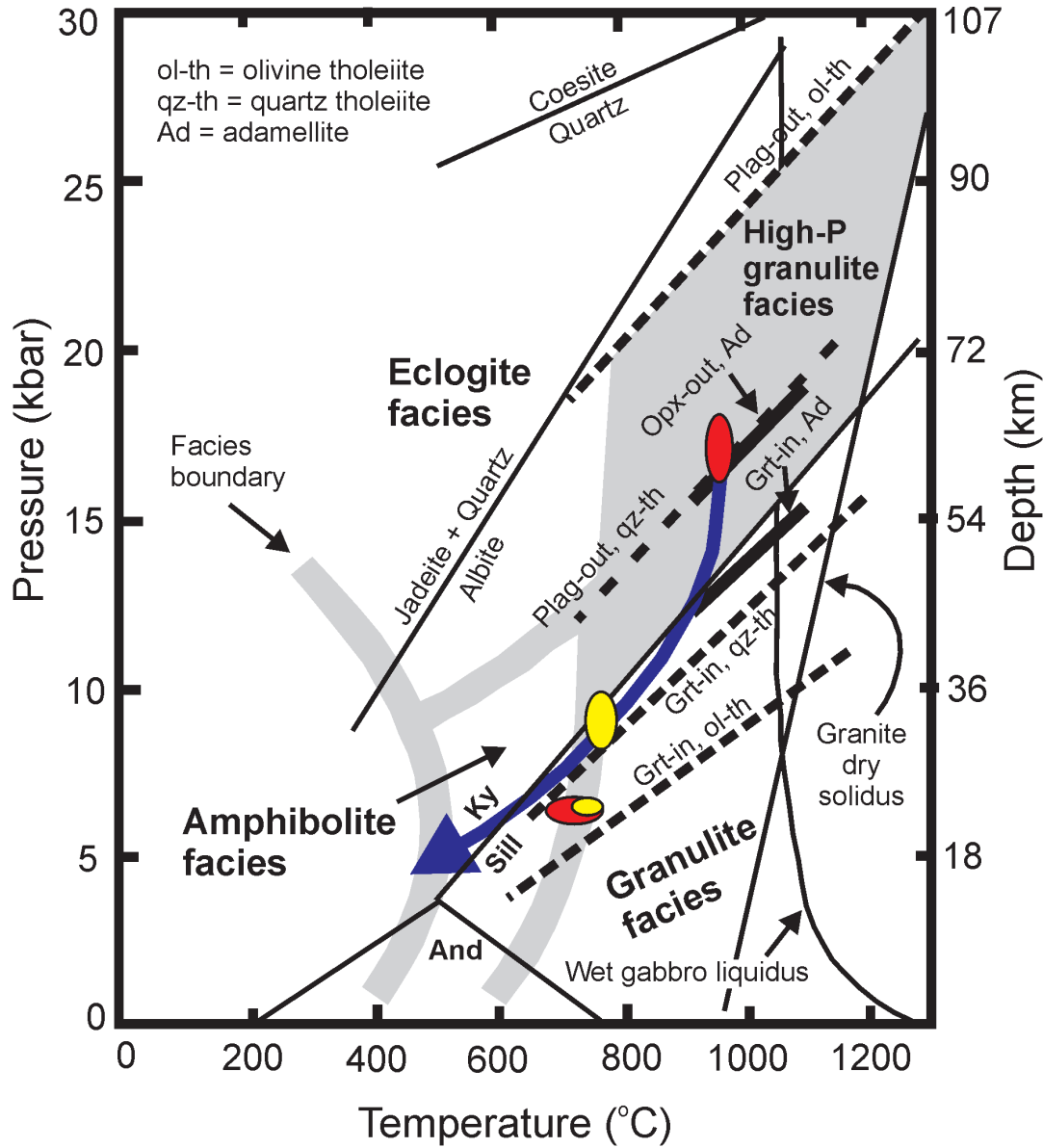
**Fig. 7.** Chemical zonation of garnet in sample EO182. alm-almandite, pyr-pyrope, spessartite, gro-grossular.

Using compositions of minerals from two polished thin sections of sample EO182 (EO182a and EO182b) and in combination with geothermometers and geobarometers, metamorphic P and T conditions of each metamorphic stage are calculated. Related geothermometers and geobarometers are garnet – clinopyroxene – plagioclase – quartz geobarometers (Eckert et al., 1991; Newton and Perkins, 1982), garnet – orthopyroxene – plagioclase – quartz geobarometer (Bhattacharya et al., 1991), garnet – clinopyroxene geothermometers (Ellis and Green, 1979; Krogh, 1988; Powell, 1985; Ravna, 2000) and garnet – orthopyroxene geothermometer (Harley, 1984). The results are presented in Fig. 8. The paragenesis garnet-clinopyroxene-plagioclase-quartz

of sample EO182a yielded pressure conditions between 16 and 18 kbar and temperatures from 930 to 960 °C (Fig. 8a), which lies in the field of high-pressure granulite facies (O'Brien and Rötzler, 2003) or in the eclogite-high-pressure granulite facies (E-HPG) field of Brown (2014). The retrogression paragenesis of EO182a yielded pressures from 6.2 to 7.0 kbar and temperatures of 660 to 750 °C (Fig. 8b), typical of the transition granulite to amphibolite facies. Sample EO182b gave intermediate results with the higher and lower pressure ranges of 8-10 and 6.3-6.5 kbar, respectively, and temperatures varying from 790 to 700 °C (Fig. 8c, d), also in the lower pressure limit of the high-pressure granulite field (Fig. 9) to transition of normal granulite to amphibolite facies. Pressure-temperature results of these two thin sections plot on a clockwise retrograde P-T path (Fig. 9), consistent with the reaction textures of the granulites.



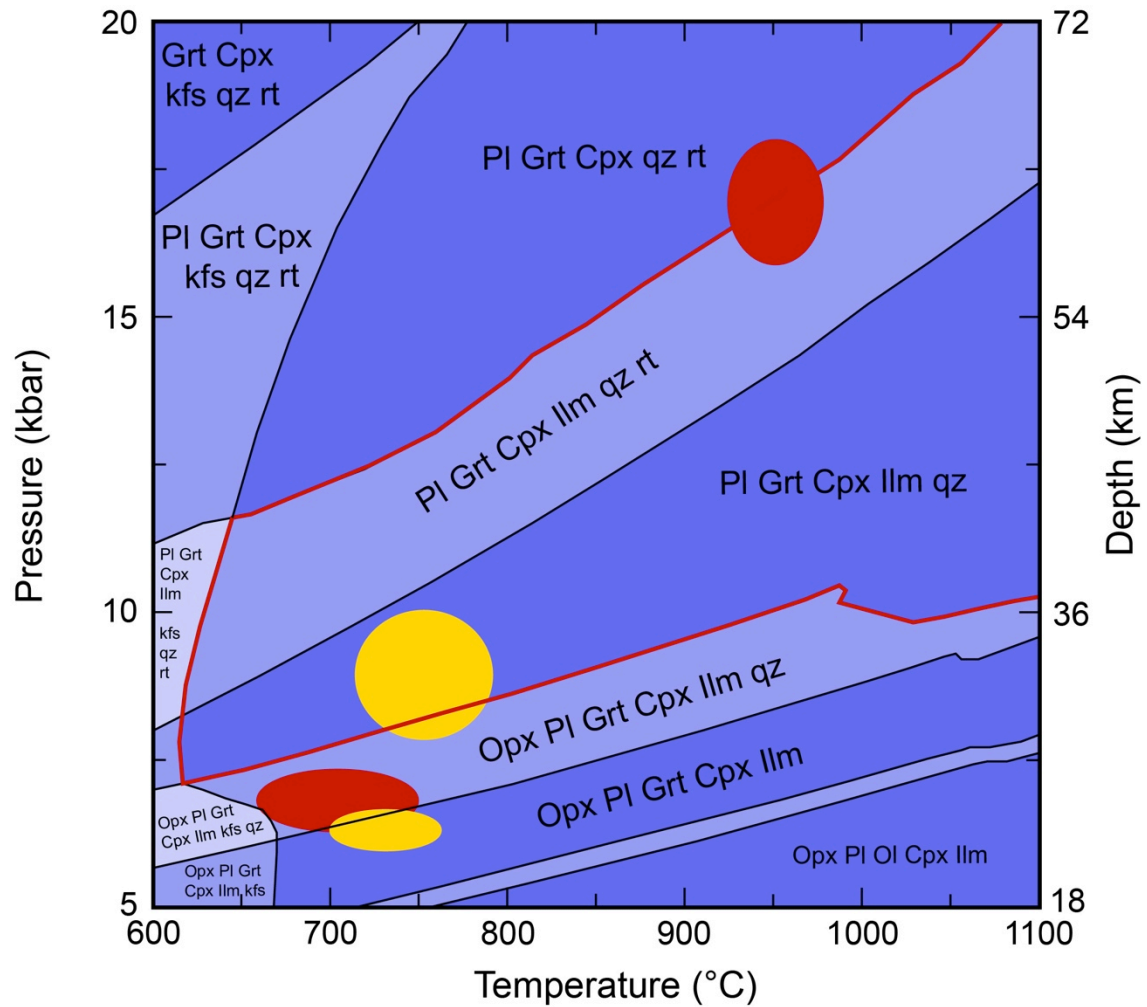
**Fig. 8.** Calculated P-T conditions of the mafic granulite EO182. (a) and (b) correspond to the high-, and low-pressure PT conditions of mineral reaction texture of thin section EO182a, respectively; (c) and (d) same for thin section EO182b.



**Fig. 9.** P-T path of the mafic granulite samples plotted in the metamorphic facies diagram of O'Brien and Rötzler (2003). Red and yellow ellipses are PT conditions for samples EO182a and EO182b, respectively (see text for details). The blue thick arrow indicates a possible clockwise retrograde path for the mineral reaction textures.

#### 4.2. Metamorphic conditions: Pressure-temperature pseudosections

Pressure-temperature pseudosections were calculated for sample EO182 bulk rock (Fig. 10). The phase field of Pl + Grt + Cpx + Ilm (red outline in Fig. 10) best represents the observed peak mineral assemblage. These pressure results overlap with 16-18 kbar obtained by thermobarometry for EO182a and with 8-10 kbar for EO182b (Fig. 8a). However, as there is a large uncertainty in the conventional thermobarometry for the same rock sample, the highest-pressure estimate may possibly be somewhat lower due to the absence of rutile in thin section. Thermobarometry for the lower-grade assemblage plot within the Opx stability field and indicate retrograde decompression and cooling.



**Fig. 10.** Pseudosection for the bulk rock composition of sample EO182. Red outline marks the field with the observed granulite facies assemblage. Colours of the phase

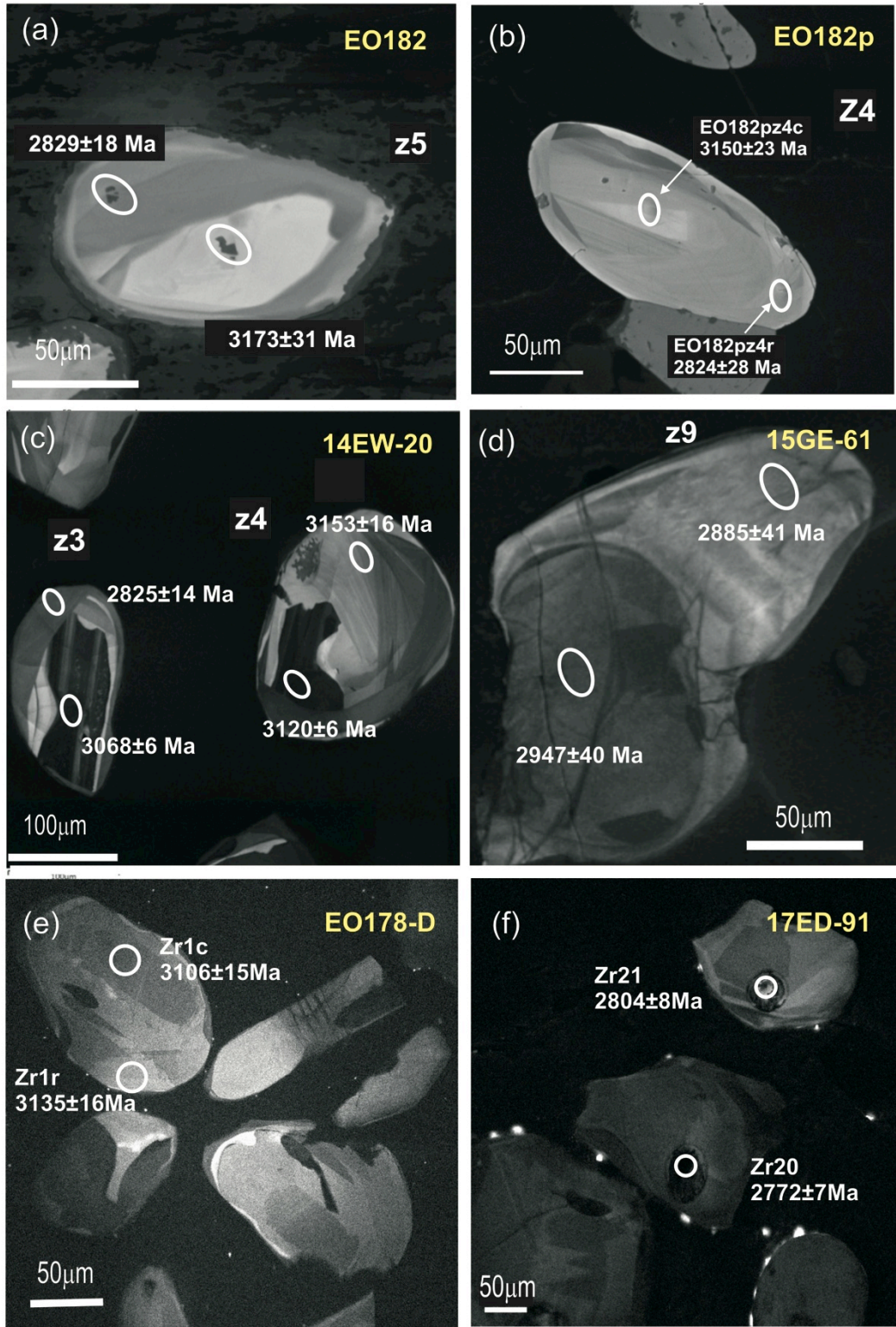
fields represent the degree of variance ranging from four phases (darker blue) to seven phases (pale blue). Red (EO182a) and yellow (EO182b) ellipses represent estimates obtained from thermobarometry for high and low P-T conditions. Mineral abbreviations: Cpx - clinopyroxene; Grt - garnet; Ilm - ilmenite; Ol - olivine; Opx - orthopyroxene; Pl - plagioclase; kfs - K-feldspar; qz - Quartz; rt - rutile (capitalized: solid solutions; lowercase: pure phases). Bulk composition of sample EO182 in wt.% is: SiO<sub>2</sub>=44.92, TiO<sub>2</sub>=2.604, Al<sub>2</sub>O<sub>3</sub>=14.49, Fe<sub>2</sub>O<sub>3t</sub>=20.30, MnO=0.308, MgO=5.25, CaO=10.04, Na<sub>2</sub>O=1.69, K<sub>2</sub>O=0.14, P<sub>2</sub>O<sub>5</sub>=0.478, LOI=-0.38, Total=100.22. Starting composition in wt% (normalized to 100) for the system TiKNCFMASO is: SiO<sub>2</sub>=46.0, TiO<sub>2</sub>=2.7, Al<sub>2</sub>O<sub>3</sub>=14.8, FeO<sub>t</sub>=18.7, MgO=5.4, CaO=10.3, Na<sub>2</sub>O=1.7, K<sub>2</sub>O=0.1, O<sub>2</sub>=0.2.

#### **4.3. Zircon SHRIMP and LA-ICP-MS U-Pb geochronology, trace elements geochemistry and Hf isotopes**

Five samples were collected for SHRIMP and LA-ICP-MS U-Pb zircon age dating, of which one is high-pressure (HP) mafic granulite (EO182), three are associated gneisses (14EW-20, 15GE-61, and EO178-D), and one trondhjemitic sheet (17ED-91) intrusive in the HP mafic granulite. Zircon inclusions in garnet from a polished slab of sample EO182 were drilled to make another sample (EO182p) for SHRIMP age dating. The results are shown in Table S3 (supplemental files). Zircon grains from the HP mafic granulite EO182p were also analysed for rare earth elements (REE) and other trace elements (Table S4 - Supplemental files). Concordant zircon grains from HP mafic granulite EO182 and leucodioritic gneiss EO178-D were also analysed for Hf isotopes; the results are shown in Table S5 (Supplemental files).

Cathodoluminescence images and spot ages of representative zircon grains of the dated samples are shown in Fig. 11.





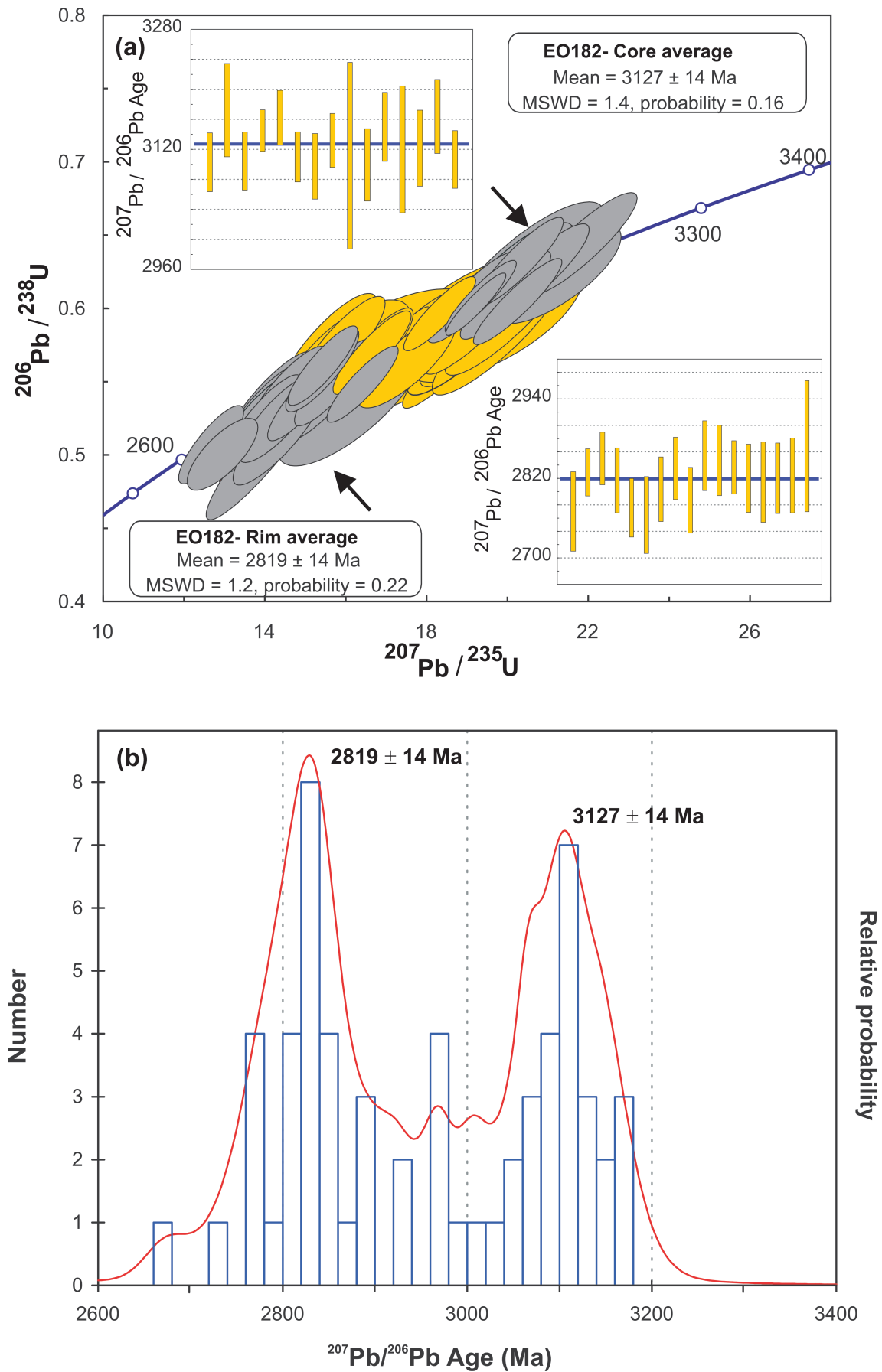
**Fig. 11.** Cathodoluminescence images and spot ages of selected zircon grains.

#### 4.3.1. Sample EO182 - High-pressure mafic granulite - SHRIMP

This sample is a massive, garnet-rich mafic granulite. The youngest seventeen analyses (Table S3) of zircon grains target visible rim zones. These analyses yielded a

population at  $2819 \pm 14$  Ma (MSWD = 1.2, probability = 0.22), which is considered the best estimate of the age of the rims (Fig. 12a) grown during high-grade metamorphism as discussed in the following section. The oldest and less abundant population in Fig. 12a comprises fifteen analyses of zircon core (e.g. Fig. 11a) with a mean  $^{207}\text{Pb}/^{206}\text{Pb}$  age of  $3127 \pm 14$  Ma (MSWD = 1.4, probability = 0.16) that represents the best estimate of the crystallization age of the protolith. A histogram with all ages is shown in Fig. 12b where age peaks of core and rim analyses are highlighted. The intermediate concordant ages are interpreted as Pb loss caused by incomplete solid-state recrystallization (e.g. Vavra et al., 1996; Hoskin & Black, 2000; Möller et al., 2002) during the 2819 Ma event.





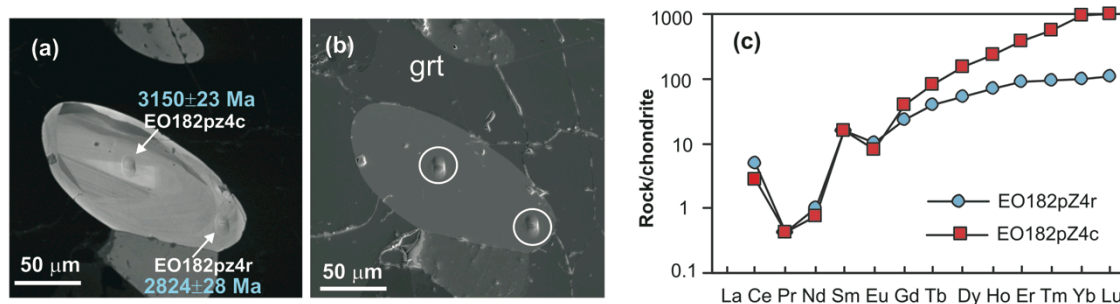
**Fig. 12.** SHRIMP U-Pb zircon ages for mafic granulite EO182. a) Concordia plot showing age averages for core and rim zircons (grey ellipses); b) SHRIMP  $^{207}\text{Pb}/^{206}\text{Pb}$

age histogram for metamorphic rims (2819 Ma) and older cores (3127 Ma) of zircon grains from sample EO182 and EO182p. The intermediate concordant ages are interpreted as Pb loss caused by incomplete solid-state recrystallization (e.g. Vavra et al., 1996; Hoskin & Black, 2000; Möller et al., 2002) during the 2819 Ma event.

#### **4.3.2. Sample EO182p -High pressure mafic granulite - SHRIMP**

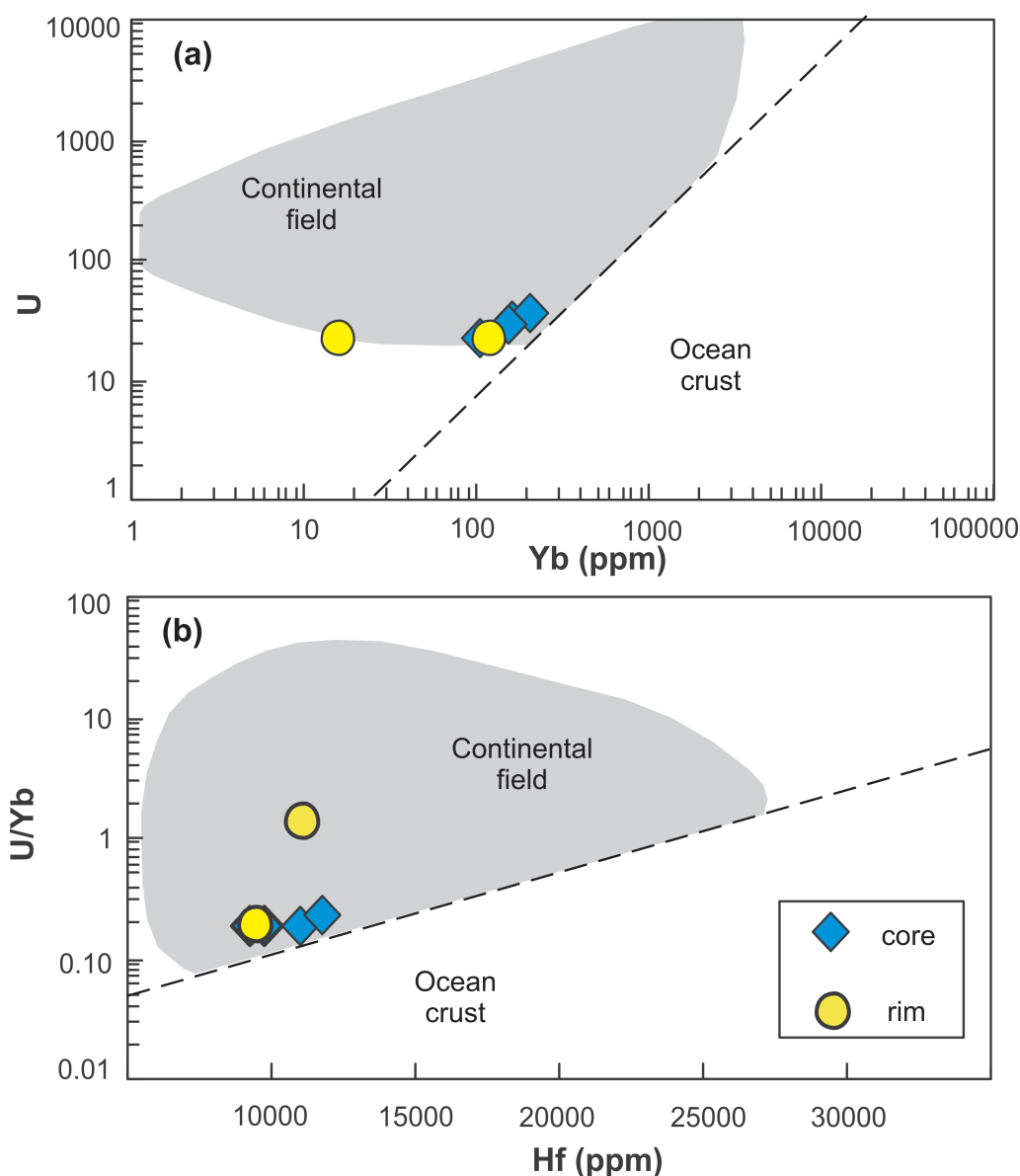
SHRIMP analyses were performed on a polished slab of sample EO182, here named sample EO182p. A few zircon grains occur as inclusions in garnet. These grains and their host garnets were drilled and mounted in epoxy resin for SHRIMP age dating (Table S3; supplemental files) and LA-ICP-MS trace element analysis. As shown in Fig. 13a, the ages of the zircon core and rim spots were  $3150 \pm 23$  Ma and  $2824 \pm 28$  Ma, respectively. Although less precise due to limited data, within error limits, these ages replicate the average ages found above for sample EO182, i.e.  $3127 \pm 14$  Ma on zircon cores, and  $2819 \pm 14$  Ma on zircon rims.

Representative trace element data for some zircon grains included in garnet are shown in Table S4 (supplemental files). The location of the SHRIMP and LA-ICP-MS spots analysis are indicated in Fig.13a and Fig.13b, respectively, and the chondrite-normalized rare earth element (REE) patterns are shown in Fig. 13c. The REE patterns of rim and core spots differ significantly. The rim REE patterns show a lower heavy-REE abundance than those of the cores, a typical signature of zircon growth in equilibrium with another heavy-REE-rich mineral (e.g. Rubatto, 2002), which in this case is garnet. Therefore, we consider that the ca. 3130 Ma growth of zircon was in the absence of garnet, whereas the ca. 2820 Ma is the time of zircon growth in the presence of garnet, and by inference this is the age of the high-pressure metamorphic event.



**Fig. 13.** Representative zircon grains as inclusions in garnet in sample EO182p. a) Cathodoluminescence image of a zircon core and rim with identification of the SHRIMP spots and  $^{207}\text{Pb}/^{206}\text{Pb}$  ages; b) SEM backscatter image of the same zircon grain in (a) showing the SHRIMP (shallow pits) and the LA-ICP-MS analysed spots (white circles); c) Chondrite-normalised (McDonough and Sun, 1995) rare earth element patterns for the zircon grain in (b) showing the higher heavy rare earth element (HREE) abundances in the core spot (EO182pZ4c) than in the rim spot (EO182pZ4r).

On the basis of trace element data on zircon grains from oceanic gabbros and continental rocks, Grimes et al. (2007) suggested that the U vs. Yb, U/Yb vs Hf or Y plots can distinguish zircon crystallized in magmas emplaced in either tectonic settings. As shown in Fig. 14, the U vs. Yb and U/Yb vs Hf plots indicate that zircons of the HP mafic granulites may have crystallized from a magma emplaced in continental settings.



**Fig. 14.** Trace elements in zircon with fields of gabbros from oceanic crust and continental rocks after Grimes et al. (2007). Data for zircon from the HP mafic granulites plot all in the continental field.

#### 4.3.3. Sample 14EW-20 - Host quartz-leucodiorite gneiss - SHRIMP

Ten zircons were analysed and sixteen of twenty analyses yielded concordant dates, which are spread from 2.83 to 3.13 Ga (Fig. 15a, Table S3). Five analyses plot in a single population with a mean  $^{207}\text{Pb}/^{206}\text{Pb}$  age of  $3125 \pm 15$  Ma (MSWD=1.5, probability = 0.20) (Fig. 15a). These analyses are from cores of complex zircons and this age may represent the crystallization age of the protolith. There are also two

younger dates at 2.82 and 2.83 Ga from rims (Fig. 15a), which agree with the ~ 2.82 Ga age of the metamorphism of the area as determined from samples EO182 and EO182p. Furthermore, there are nine intermediate concordant dates ranging from 2.92 to 3.10 Ga (Fig. 15a), which are interpreted as Pb loss caused by incomplete solid-state recrystallization (e.g. Vavra et al., 1996; Hoskin & Black, 2000; Möller et al., 2002) during the 2819 Ma event.

#### **4.3.4. Sample 15GE-61 - Host leucodiorite gneiss - SHRIMP**

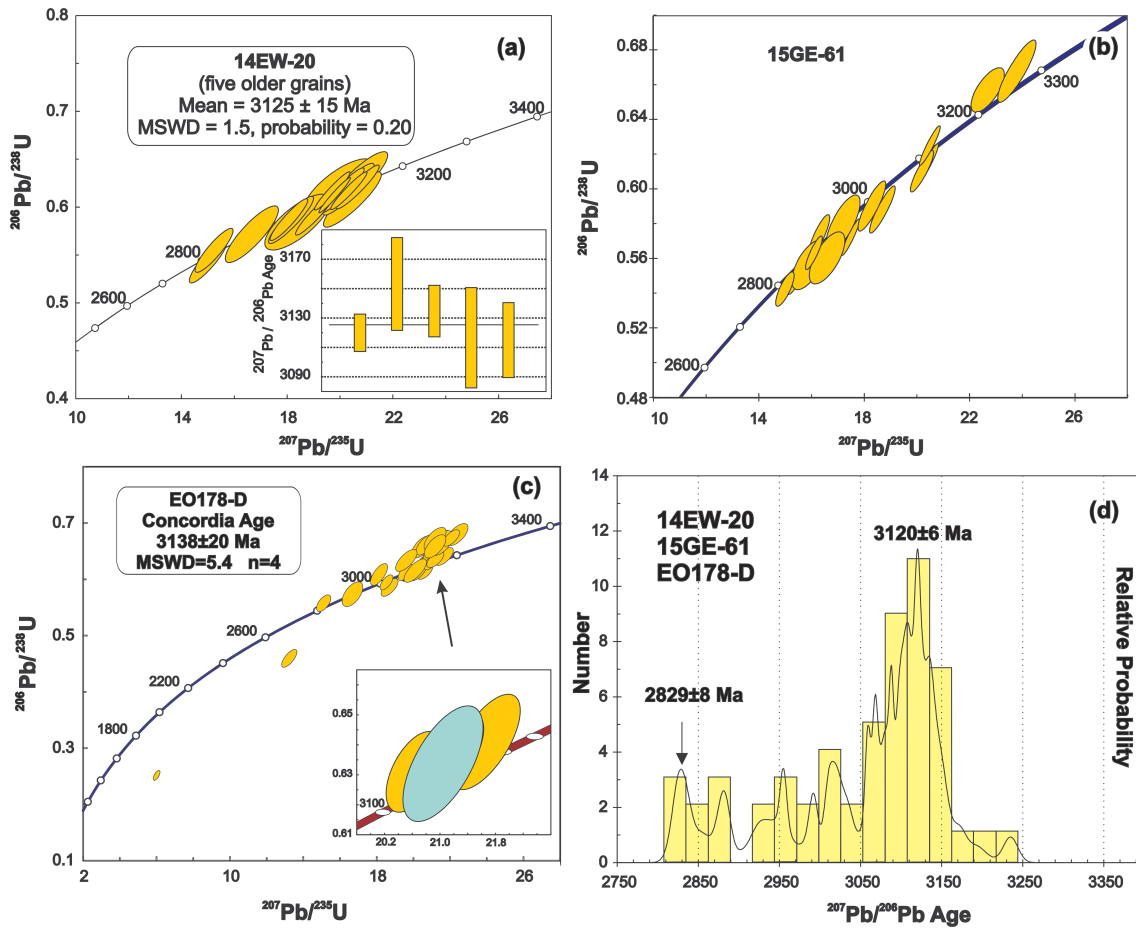
Twenty analyses, performed on fifteen zircons, yielded concordant dates ranging from 2.83 to 3.26 Ga (Fig 15b; Table S3). There are five dates with ages from 2.83-2.88 Ga (Table S3; e.g. 15GE61z12), which broadly agree with the age of the metamorphism of the area (i.e. ~2.82 Ga from sample EO182). There are also three dates at 3.11-3.12 Ga (Table S3; e.g. 15GE61z5c), which may be considered the crystallization age of the protolith, and which match the other samples. Moreover, there are three older dates at 3.19-3.26 Ga, which may represent xenocrysts, but have not been observed in the other samples. Apart from this, there are eight intermediate concordant dates between 2.94 and 3.06 Ga, which are interpreted as Pb loss caused by incomplete solid-state recrystallization (e.g. Vavra et al., 1996; Hoskin & Black, 2000; Möller et al., 2002) during the 2819 Ma event.

#### **4.3.5. Sample EO178-D - Host leucodiorite gneiss - LA-ICP-MS**

Twenty-five zircon grains were analysed and twenty-two of thirty-two analyses give concordant ages ( $\pm 5\%$  discordant; Table S3). Zircon CL images show sector field zoning or unzoning (Fig. 11e); some grains show dark cores and bright rims. Thirteen, less than 2% discordant zircon core analyses yielded an upper intercept date of  $3104 \pm$

24 Ma (MSWD = 2.4; probability = 0.01) and four grains yielded a concordia age of 3138±20 Ma (Fig. 15c). Some grains yielded ca. 2.99 Ga rim dates and 3.06 Ga cores (Table S3), which may be due to Pb loss. Only one zircon core analysis (z5, Table S3) yielded a date of 2823 Ma comparable with the age of metamorphism observed in mafic granulite EO182.

Combining the concordant data of the host gneisses (14EW-20, 15GE-61, and EO178-D), two endmember age populations at 2.81-2.85 Ga and 3.08-3.15 Ga (Fig. 15d) can be distinguished. These populations can be also recognized in the HP mafic granulite (Fig. 12). The youngest ages at 2.82-2.83 Ga is considered to represent the age of metamorphism of this area. The oldest ages at 3.11-3.12 Ga, with an age cluster at 3120±6 Ma may be the crystallization age of the protolith. However, intermediate concordant ages could be due to Pb loss caused by incomplete solid-state recrystallization (e.g. Vavra et al., 1996; Hoskin & Black, 2000; Möller et al., 2002) during the 2819 Ma event.

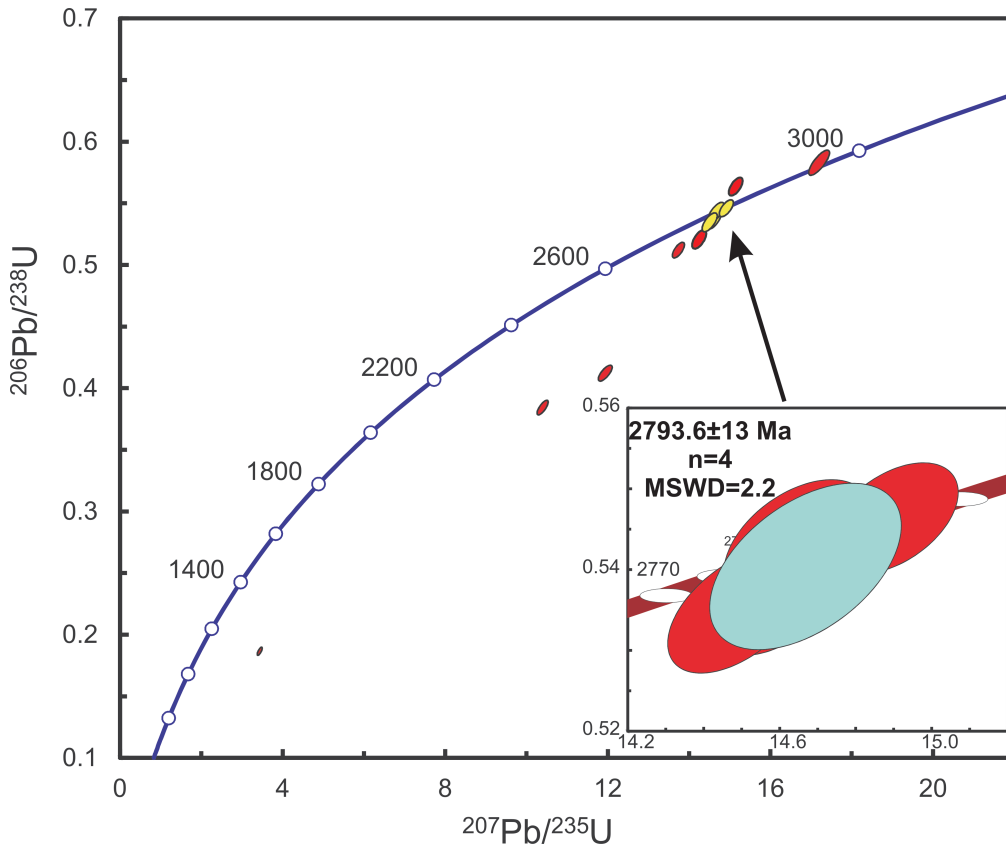


**Fig. 15.** SHRIMP and LA-ICP-MS U-Pb ages of diorite-leucodiorite gneisses associated with the HP mafic granulite. a) Concordia plot of zircon grains from sample 14EW-20 and a weighted average age of  $3125 \pm 15$  Ma for the five oldest grains; b) Concordia plot for zircon grains from sample 15GE-61; c) Concordia plot for all zircon grains of sample EO178-D with concordia age shown in inset for the most concordant grains ( $<1\%$  discordant); d)  $^{207}\text{Pb}/^{206}\text{Pb}$  age histogram for the combined ages of samples 14EW-20, 15GE-61, and EO178-D.

#### 4.3.6. Sample 17ED-91 - Trondhjemite sheet intrusive in mafic granulite - LA-ICP-MS

Twenty-one zircon grains were analysed and eleven had to be discarded due to high common Pb. From the remaining eleven zircon analyses (Table S3) only eight analyses give concordant ages ( $<5\%$  discordant), of which one grain was considered

inherited (2927 Ma). The remaining seven analyses gave an upper intercept age of  $2797 \pm 5$  Ma (MSWD = 1.12; probability = 0.34) and four grains less than 1% discordant gave a concordia age of  $2793.6 \pm 13$  Ma (Fig. 16), which is considered the crystallization age of the trondhjemite sheets.



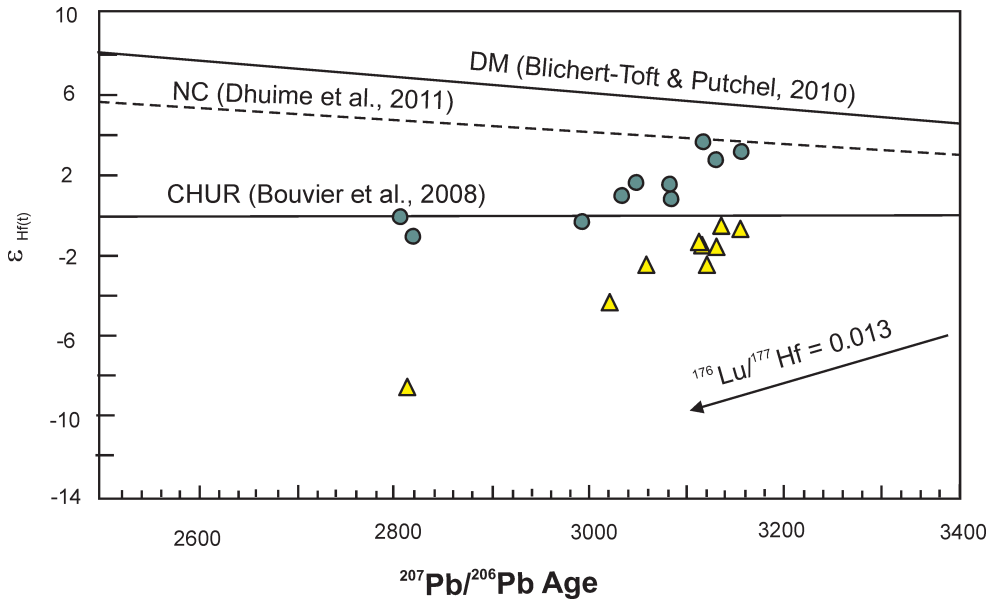
**Fig. 16.** LA-ICP-MS zircon U-Pb concordia age of 17ED-91 trondhjemite sheet intrusive in the HP mafic granulite.

#### 4.3.7. Hf isotopes results - LA-ICP-MS

Zircon grains from two samples were analysed for Hf isotopes, namely the mafic granulite EO182 and leucodioritic gneiss EO178-D. The results are shown in Table S5. Depleted mantle (Blichert-Toft et al., 2010) and new crust (Dhuime et al., 2011) evolution are shown with the initial  $\epsilon_{\text{Hf}(t)}$  values of the mafic granulites and leucodioritic gneisses in Fig. 17. The Hf data come from the same location on



zircon grains that yielded concordant age data. The studied rocks show distinct  $\epsilon_{\text{Hf}(t)}$  values with mostly positive values (relative to CHUR) for the mafic granulite and negative values for the associated dioritic gneiss (Fig. 17). Field relationships (layering) indicate that the two rock types are co-magmatic, and yet they show different, although paralleling Lu-Hf isotopic evolution trends.



**Fig. 17.** Hf isotopes data for the Uauá HP mafic granulite EO182 (circle) and associated leucodioritic gneiss EO178-D (triangle). Two almost parallel trends are depicted for the studied samples, wherein the mafic granulite shows a clear juvenile component; the analyses close to 3120 Ma are representative of the protoliths age, those at 2.82 Ga are from metamorphic zircon rims, and intermediate ones are mixed ages due to Pb loss caused by incomplete solid-state recrystallization (e.g. Vavra et al., 1996; Hoskin & Black, 2000; Möller et al., 2002) during the 2.82 Ga event. All spot analyses were on locations that gave concordant analyses. Archaean upper crust growth  $^{176}\text{Lu}/^{177}\text{Hf}$  value (0.013) after Condie et al. (2005).

## 5. Discussion

## 5.1. Metamorphic conditions and global Archaean high-pressure granulites

The Uauá mafic granulite occurs as sheet-like bodies (locally with discrete igneous layering), or lenses in structural conformity with ca. 3120 Ma-old leucodiorite to quartz-diorite gneisses and ca. 2800 Ma trondhjemite sheets. The mafic granulite has a relic high-pressure (HP) granulite facies assemblage of garnet-clinopyroxene with minor quartz, plagioclase and ilmenite. This paragenesis reached the HP metamorphic equilibrium about 16-18 kbar and 950°C ca. 2820 Ma ago, according to our thermobarometry and pseudosections calculations and the SHRIMP U-Pb age dating of zircon rims that have grown in equilibrium with garnet. Garnet porphyroblasts have a corona of orthopyroxene-plagioclase symplectites, a textural relationship, which, when combined with minor hornblende in the symplectites and along the granulite foliation, defines retrogression from HP granulite facies through normal granulite facies to transition to amphibolite facies conditions of 6.2 to 7.0 kbar and 660 to 750 °C.

High-pressure metamorphic rocks in the Archaean are not common. The oldest high-pressure metamorphic rock in upper crustal greenstone belts is a 3200 Ma meta-volcanic amphibolite from the Barberton belt in South Africa with a HP condition of 12-15 kbar at a relatively low temperature of 600-650°C (Moyen et al., 2006). Another example can be found in the Belomorian province of Russia where high-pressure granulite (retrogressed eclogite) lenses in gneisses have been described as Archaean but the metamorphic age is contentious. Accordingly, Volodichev et al. (2004), Mints et al. (2010, 2014), Dokukina et al. (2012), and Li et al. (2015) suggested from zircon dating that the HP metamorphism was Archaean (between 2870 and 2710 Ma). However, on the basis of Lu-Hf isochron dating of eclogite garnet (Herwartz et al., 2012, Yu et al., 2019), garnet-whole rock Sm-Nd isochron and zircon U-Pb of garnetites (Mel'nik et al., 2013), U-Pb zircon dating with omphacite + garnet inclusions (Imayama et al., 2017), and metamorphic zircon rims surrounding Archaean magmatic zircon cores (Skublov et

al. 2010, 2011) these authors provided evidence for the high-grade metamorphism at ca. 1900-1920 Ma.

Many other high-pressure granulites have yielded ages that straddle the Neoarchaeon-Palaeoproterozoic transition, such as meta-pelitic granulites in the Limpopo belt (ca. 2700 Ma; Van Reenen et al., 1987; Droop, 1989), mafic granulites in the Athabasca terrane, Canada (ca. 2550 Ma; Baldwin et al., 2006; Williams et al., 2009), the North China Craton (2520 Ma at Yinshan, Wang and Guo, 2017; 2510 Ma at Hebei, Kwan et al., 2016; 2500 Ma at Jianping, Wei et al., 2001; 2473 Ma at Jiaodong, Liu et al., 2015), the Lewisian in Scotland (ca. 2480 Ma, Sajejev et al., 2013), and in India (2780 Ma in the Bundelkhand craton, Saha et al., 2011; 2541 Ma at Sittampundi, Sajejev et al., 2009; ca. 2490 Ma in the Salem block, Anderson et al., 2012; ca. 2460 Ma in the Nilgiri Block, Samuel et al., 2015). As such, the Uauá HP mafic granulite, with the age of ca. 2820 Ma is the World's second oldest HP mafic granulite, and possibly records significantly higher pressures and temperatures than the oldest.

## **5.2. Crustal thickening and geodynamic scenario**

Thermodynamic derived pressure estimates for equilibrium of metamorphic assemblages can be linked to the lithostatic pressure, which in turn can be directly related to depth (e.g. Jolivet et al., 2003). This general assumption of metamorphic petrology has been challenged recently due to the suggestion that tectonic deformation (differential stress, non-hydrostatic stress) can produce larger pressures than lithostatic pressure (e.g.; Schamalholz and Podladchikov, 2014; Wheeler, 2014; Tajcmanová, 2015; Reuber et al., 2016). However, for lower crustal rocks where permeability and porosity may be exceedingly small (Ingebritsen and Manning, 2002), the mean stress is not far from the vertical stress in value, and the pressure versus depth interpretation applies (Wheeler, 2014). Moreover, on a recent account about equilibrium in non-

hydrostatic metamorphic systems, Powell et al. (2018) concluded that calculations that incorporate the equilibrium thermodynamics of non-hydrostatically stressed minerals yield results very similar to those assuming hydrostatic stress, and as such the estimated pressure may be related to the depth of metamorphism.

In this study, the pressure estimates using the thermobarometers of Eckert et al. (1991) and Newton and Perkins (1982) indicate pressures of 16-18 kbar for the high-pressure mineralogy (Fig. 8-9), which was corroborated by quantitative phase diagrams (pseudosections - Fig. 10). This pressure range suggests a crustal thickness of about 60 km (Fig. 9, 10), which in the Phanerozoic is found in accretionary and collisional orogens. The clockwise retrogression path of the studied granulites from high-pressure granulite facies through normal granulite facies to amphibolite facies (Fig. 9) is also consistent with crustal thickening in collisional orogens followed by tectonic exhumation (e.g. Carswell and O'Brien, 1993; Barker, 1998; Liu et al., 2013).

How thick was the crust in the Archaean and how it became thick to form the first cratons and supercratons; whether by subduction, collision, underplating, or intracontinental thrusting, it is a subject of current debate (e.g. Smirnov et al., 2013; Brown 2014; Sizova et al., 2015; Tang et al., 2016; Johnson et al., 2017). However, there is growing evidence that plate tectonics became global between 3.0 Ga and 2.5 Ga, and could produce a thick lithosphere required to form Archaean high-pressure granulites and eclogites (e.g. Van Kranendonk et al., 2007; Burke, 2011; Polat, 2013). For example, Shirey and Richardson (2011) reported the first occurrence of ca. 3.0 Ga eclogitic garnet with basaltic Re/Os ratios (2-30 as opposed to peridotitic ratios of 0.01-0.03) as inclusions in diamonds; Dhuime et al. (2012) recognized the increase of recycling of crust into the mantle by 3.0 Ga probably through subduction at convergent plate margins; Moyen et al. (2006) suggested that the high-pressure and relatively low temperature of 3200 Ma amphibolite from the Barberton belt, in Africa, supported the

operation of subduction-driven processes in the Archaean; and Brown (2006) suggested that the first appearance of ultrahigh-temperature (UHT) and medium-temperature eclogite–high-pressure (EHP) granulite metamorphism in the Neoarchaean marks the onset of plate tectonics ("Proterozoic plate tectonics" to distinguish from modern plate tectonics that is marked by ultrahigh-temperature and ultrahigh-pressure metamorphism). Evidence for the punctuated operation of plate tectonics in the Early Archaean has been well reported (e.g. Komiya et al., 1999, 2015; Polat et al., 2009; O'Neill et al., 2013), and it is currently increasingly accepted that some kind of global plate tectonics, either of Proterozoic-type regime (Brown, 2006; 2009; 2014; Brown and Johnson, 2018) or modern-type regime was in wide operation between 3.0-2.5 Ga. The shallow dipping, 2820 Ma-old Uauá high-pressure mafic granulites and their ca. 3120 Ga protoliths formed approximately during this period, and they may shed some lights into models of crustal evolution in the Archaean.

The following petrogenetic constraints suggest that the igneous protoliths of the Uauá HP mafic granulites and associated leucodiorite gneiss were emplaced in a continental tectonic setting. Firstly, the Uauá high-pressure mafic granulite crops out as sheets interleaved with leucodiorite- and quartz-diorite gneisses. These rocks grade into each other through a layered zone (Fig. 5a), which indicates they belong to a plutonic igneous complex. The igneous protoliths of these rocks have a similar age ( $3127 \pm 14$  Ma for the mafic granulite and  $3120 \pm 6$  Ma for the leucodiorite gneiss) but the leucodiorite gneiss contain older inherited zircon grains (3.19-3.26 Ga). Secondly, U, Yb, and Hf contents in zircon grains from the mafic granulite (Fig. 13) indicate that zircon grew in continental settings rather than in the ocean crust. Thirdly, Hf isotopes in zircon yield positive  $\epsilon_{\text{Hf}(t)}$  values for the mafic granulite and negative values for the associated leucodiorite gneiss; zircon positive  $\epsilon_{\text{Hf}(t)}$  values indicate magmas derived direct from the mantle, whereas negative values imply more complex processes, such as crustal

contamination of mantle magmas and reworking of older crust (e.g. Dhuime et al., 2012; Iizuka et al., 2017).

Given that field relationships (layering) show that the protoliths of the two rock types belong to a single igneous complex, then there are three possibilities to explain the observed contrasting zircon Hf isotope signatures. The first possibility is that the emplacement of the parental, mantle-derived basic magma of the mafic granulite into an older continental crust was accompanied by partial melting of this older crust to produce the dioritic magma that was subsequently mixed with the resident basic magma. The second possibility is that the emplacement of a single parental basic magma in older continental crust was followed by fractional crystallization of this magma to produce the more evolved leucodiorite, which in turn would interact/contaminate with older rocks. The third possibility is that a juvenile basic magma emplaced into a leucodioritic magma chamber that was contaminated with older continental crust. The latter possibility is preferred for many reasons: i) the occurrence of continental crust older than the protolith age (ca. 3.12 Ga) of the studied rocks in the Uauá area is very much likely because three zircon grains with ages of ca. 3.2-3.3 Ga were found in the leucodiorite gneiss 14EG-61 (Table S3); and ii) the igneous protoliths of the mafic granulite and associated diorite gneiss were arguably derived from two magma types as suggested by their distinct Hf isotope signatures in zircon (Fig. 17). Having concluded that the protoliths of the mafic granulite and associated leucodiorite gneiss might have emplaced in an older continental crust, another relevant issue to be discussed is the relatively shallow-dipping foliation observed in the studied rocks.

Shallowly dipping foliation in granulite facies terrains has been described in both contractional (e.g. Dumond et al., 2010; Masquelin et al., 2017) and extensional (Harris et al., 2002; Arango et al., 2013) tectonic settings. Foliation of the Uauá HP mafic granulite and associated leucodiorite gneiss dips 5-25° to NE-SE with quartz or



feldspar stretching lineation plunging to NE (Fig. 3). Locally, more deformed leucodiorite gneiss shows subhorizontal, decimetre-thick mylonite bands with top-to-SW kinematics (Fig. 5d) suggesting that contraction was probably active during and after the high-pressure granulite metamorphism. Exhumation of the HP rocks was accompanied by extensive retrogression to normal granulite and amphibolite facies, and might have taken no more than 20 million years as indicated by intrusion of trondhjemite sheets in the retrogressed granulite/amphibolite facies rocks ca. 2800 Ma. Consequently, and using the pressure-depth relationship shown in Fig. 9, the exhumation rate from high-pressure granulite to amphibolite facies was about 2 mm/year, a number comparable to Phanerozoic exhumation velocities of subducted oceanic crust (Agard et al., 2009). We suggest that the mafic and felsic igneous protoliths of the mafic granulite and leucodiorite gneiss were deformed and metamorphosed by contraction and crustal thickening during collision of blocks/terranes not yet constrained. Later, the resulting larger continental block accreted to other blocks or cratons to form one of Earth's first supercratons by the end of the Mesoarchaeon.

Bleeker (2003) coined the name "supercratons" to include all large ancestral landmasses of Archaean age with a stabilized core that on break-up spawned several independently drifting cratons. The oldest supercraton was probably Vaalbara (Cheney, 1996) that cratonized about 3.0 Ga (Moser et al., 2001); its descendants are the Kaapvaal, Pilbara, and possibly also the Singhbhum craton in India (Kumar et al., 2017). The next supercratons in age are Sclavia and Superia, which cratonized about 2.6 Ga and 2.65 Ga (Bleeker, 2003), respectively. Brown (2006, 2007) observed that the formation of the supercratons Superia and Sclavia, and of the supercontinents Nuna, Rodina, and Gondwana was shortly preceded by periods of eclogite-high-pressure granulite metamorphism. Therefore, the Uauá terrane, with its 2820 Ma high-pressure mafic granulite occurrences, may represent a fragment of the root of a major Archaean

supercraton formed at the end of the Mesoarchaeon, or most probably at the dawn of the Neoarchaeon. Salminen et al. (2018) have recently discussed this possibility. Using magmatic age barcode comparisons, cratonization ages, paleomagnetism and age of mafic dyke swarms, Salminen et al. (2018) propose that the northern São Francisco (Uauá block), Kaapvaal and Pilbara cratons were nearest neighbours in the Mesoarchaeon, and along with other cratons (Superior, Karelia + Kola, Zimbabwe, Yilgarn, Tanzania) they all were probably part of the Archaean supercraton Supervaalbara of Gumsley (2017).

## 6. Conclusions

- 1) Uauá mafic granulite occurs as sheet-like bodies (locally with discrete igneous layering), or lenses in structural conformity with leucodiorite to quartz-diorite gneisses and crosscut by ca. 2800 Ma trondhjemite sheets.
- 2) Igneous protoliths of the mafic granulite and associated leucodiorite gneiss may belong to a single igneous complex ca. 3120 Ma old, possibly emplaced in a continental setting.
- 3) Mafic granulite has a relic high-pressure granulite facies assemblage of garnet-clinopyroxene with minor quartz, plagioclase and ilmenite. This paragenesis reached the metamorphic equilibrium about 16-18 kbar and 930-960°C ca. 2820 Ma ago.
- 4) Garnet porphyroblasts have a corona of orthopyroxene-plagioclase symplectites, a textural relationship, which, when combined with minor amphibole in the symplectites and hornblende blades oriented along the granulite foliation, defines retrogression to 6.2 to 7.0 kbar and 660 to 750 °C in the granulite to amphibolite facies transition.
- 5) Exhumation from high-pressure conditions to normal granulite and amphibolite facies has probably taken no more 20 million years at a rate of about 2 mm/year, a

number comparable to Phanerozoic exhumation velocities of subducted oceanic crust.

6) Uauá high-pressure mafic granulite, with the age of ca. 2820 Ma is the World's second oldest HP mafic granulite.

7) Crustal thickening due to blocks/cratons collision to form the high-pressure granulite possibly was in operation in the late Mesoarchaeon preceding the formation of Earth's first supercratons.

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**Appendix A. Supplementary data (tables S1-S5):** Sample location (Table S1); Mineral composition (Table S2); Zircon U-Pb data (Table S3); Zircon trace element (Table S4); Zircon Hf isotope data (Table S5);

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